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SYMPOSIUM ON RESERVOIR DATA

In February 1957 the Kuwait Branch of the Institute of Petroleum organized a symposium of papers on the general theme of the acquisition and on the analysis of reservoir data. The papers which were presented at this meeting are reproduced in the following pages, together with summaries of certain other papers and notes which were made available to the delegates.

PRELIMINARY CONSIDERATIONS OF ALBORZ OIL RESERVOIR AT QUM *

By H. FARKHAN † and J. TAVANA ‡

A CURSORY examination of the figures representing the gross quantity of residual oil lost with the abandonment of depleted reservoirs [13] will emphasize the importance of scientifically controlling petroleum reservoirs and well systems and carefully analysing all the fundamental data which may be available. The data which may be required have been grouped as follows [2]:

(1) Physical properties of the reservoir.
(2) Physical character of the reservoir fluids.
(3) Static characteristics of the rock-fluid system.
(4) Dynamic characteristics of the rock-fluid system.

It should be pointed out, however, that reservoir studies can neither be restricted to any one specific group of factors nor limited to any time or place.

For instance, it is known that the structural conditions of the reservoir, and many geological factors, may affect its behaviour.[9, 10] Similarly, it may be said that the oil reservoir control commences with the beginning of drilling operations by controlling and adjusting the essential properties of mud in such a way as not to plug off the pore system and hamper flow of oil,[11] etc. Studies of reservoir characteristics may be extended outside the reservoir proper, such as those which were carried out in the Asmari mountain and which threw considerable light on the behaviour of Masjid-i-Suleiman reservoir.[6]

It was such facts as these which led the Iran Oil Company to initiate a series of studies covering all the possible characteristics of the prospective Alborz oil pool. Attention was first directed to the limestone formation which is thought to contain commercial accumulation of oil.[12] The rocks considered are the limestones which belong to the Marine Formation of the Oligo-Miocene [5] age.

This limestone was cored in a test well located NE. of Alborz and just S. of Separ-i-Rostam (Fig 1), where it crops out as an inselberg.[4] It was also systematically sampled in Kuh-i-Nardaghi (Fig 1), S. of Qum.

The above-mentioned test well reached the top part of the Marine Formation at a depth of nearly 100 m, and the formation was continuously cored to a depth of 900 m. Cores were recovered in good condition. Bulk density, grain density, and porosity measurements were carried out on samples cut off at almost one metre intervals. A summary of these measurements is tabulated in Table I.

TABLE I

Porosity and Density Values of Marine Limestone Cores South of Separi-i-Rostam

Depth range, m	Grain density range	Porosity range, per cent
100–150	2·53–2·74	6–11·3
150–200	2·48–2·70	1–17
200–250	2·51–2·79	2·3–20
250–300	2·56–2·76	4·3–13·5
300–350	2·53–2·76	3·2–18·4
350–400	2·55–2·71	1·7–16
400–450	2·53–2·87	3·1–18·7
450–500	2·58–2·76	5·7–17·3
500–550	2·60–2·75	7·24–12·8
550–600	2·56–2·96	6·47–15·7
600–650	2·59–2·71	2·04–7·2
650–700	Not available	
700–750	2·53–2·73	1·73–7·9
750–800	2·57–3·04	1·37–9·9
800–850	2·59–2·73	2·03–8·7
850–900	2·63–3·01	1·12–6·8

These density and porosity values have been determined gravimetrically.[1] They were checked with values obtained by means of an air expansion porosimeter,[3] as well as by means of the saturation method, using kerosine as the saturating medium.[14]

A consideration of the porosity values of the limestone samples collected at Kuh-i-Nardaghi shows that

* MS received 17 June 1957. † Technical Director, Iran Oil Co. ‡ Head of Petroleum Engineering, Iran Oil Co.

they vary laterally, increasing with the decrease of the carbonate content of the limestone and its gradual change into marl.

The only reservoir fluids available for examination were the oils which flowed out of the Alborz No. 3 well and Alborz No. 5 well. The results of the tests are tabulated in Tables II and III; the change of specific gravity and viscosity of A.5 weathered crude are shown graphically on Figs 2 and 3.

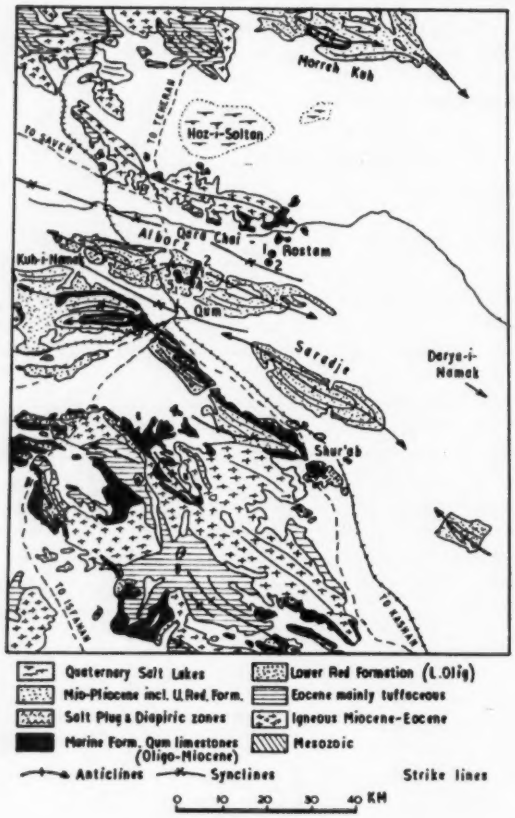

FIG 1

GEOLOGICAL MAP OF QUM AREA

Distillation, viscosity, and asphaltene content tests were carried out in accordance with the IP and the rest according to ASTM standards.

As has been mentioned elsewhere,[12] it was not possible to obtain a complete set of data on the wells drilled at the Alborz field. The following facts may be of interest.

The upper red formation,[4, 5] overlying the cap rock (evaporite) is relatively easily penetrated by the drill. However, as soon as the chemical series acting as the cap rock is reached and ordinary mud is replaced by saturated brine mud to drill through this series, which includes rock salt layers of varying thickness, con-

TABLE II

Analysis of Crude Oil from Alborz No. 3 Well at Qum

Specific gravity at 60° F	0·8354
Redwood I viscosity at 100° F	47 sec
Pour point	75° F
Water and sediment	0·2% vol
Carbon residue	2·72% wt
Sulphur	0·13% wt
Asphaltenes	0·365% wt
Chloride content	11 p.p.m.
Distillation:	
I.B.P.	94° C
Distillation to:	
75° C	—
100° C	0·5%
125° C	4·5%
150° C	10%
175° C	16%
200° C	21%
225° C	26·5%
250° C	32%
275° C	39%
300° C	45%
Volume of:	
Distillate	46%
Residue	53·8%
Loss	0·2%

TABLE III

Analysis of Crude Oil from Alborz No. 5 Well at Qum

Crude oil (drum sample taken at atmospheric pressure):

C1	Trace	Trace
C2	0·14 vol %	0·06 wt %
C3	0·52 vol %	0·32 wt %
Ist-C4	0·26 vol %	0·17 wt %
Normal C4	1·22 vol %	0·86 wt %
Ist-C5	0·92 vol %	0·69 wt %
Normal C5	1·39 vol %	1·06 wt %
C6 and higher	95·55 vol %	96·84 wt %
Specific gravity	0·8310 at 60° F	
API gravity	38·8° at 60° F	
I.B.P. on distillation	52° C	
Volume of distillate	3% at 75° C 8% at 100° C 13% at 125° C 18% at 150° C 28·5% at 200° C 33% at 225° C 38·5% at 250° C 44% at 275° C 49% at 300° C	
Total distillate	49·5% by vol	
Total residue	49·5% by vol	
Sp. gr. of distillate	0·7765 at 60° F (50·65° API)	
Sp. gr. of residue	0·8863 at 60° F (28·15° API)	
Total sulphur	0·085% by wt	
Carbon residue	1·25% by wt	
Asphaltenes (hard asphalt)	0·08% by wt	
Ash	0·03% by wt	
Sediment (by extraction)	0·005% by wt	
H_2S	Nil	
Wax content	10·8% by wt	
M.p. of wax	132° F	
Reid v.p.	5·8 p.s.i.g. at 100° F	
Viscosity, Redwood I	37 sec at 100° F	
Kinematic viscosity	4·57 cS at 100° F	
Pour point (upper)	70° F	
Total acidity	0·024 mg KOH/g	
Water	0·06% by vol	
B.S. & W.	0·2% by vol	
Salt content	12·3 lb/1000 brl	

siderable stuck-pipe trouble was encountered. As an instance the Alborz No. 4 well may be mentioned. The drilling of this well began with a 18½-inch hole using ordinary mud about 75 lb/cu. ft. and having a 1500–1000 Marsh funnel viscosity of 32–37 sec. The casing, 13⅜-inches o.d., 68 lb/ft. J.55, was set at

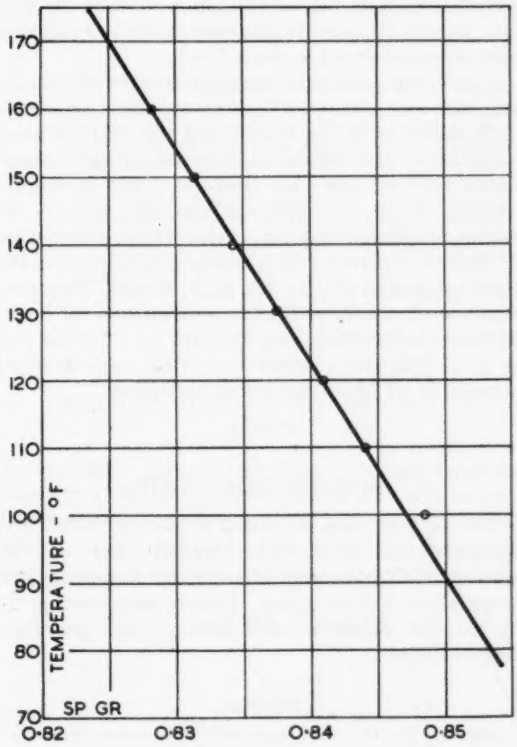

FIG 2

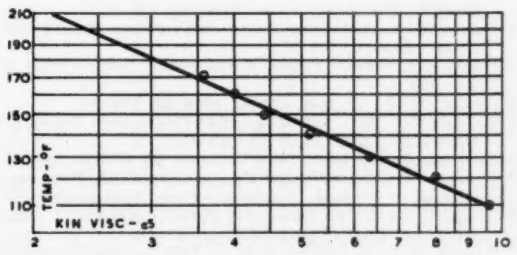

FIG 3

151 m. Thereafter the drilling of the well was continued to the chemical series, *i.e.* to a depth of over 2000 m with an uncased hole of almost the same depth showing no sign of caving and causing no difficulty. However, shortly after the sweet water mud was replaced by the brine mud, the drill pipe got stuck and pieces of rock from the upper part of the long uncased hole began falling on the bit. The hole had to be abandoned because the drilling tool, which was frozen at a depth of over 2500 m, could not be pulled free.

The next well, Alborz 5, was drilled to the evaporite. But before penetrating the chemical series, where the use of heavy brine mud is imperative, a second string of casing was inserted in the well to protect the upper formation from the disintegrating effect of the brine infiltration. The abnormal formation pressures of the evaporite series, which were considered mainly responsible for freezing the drill pipe in Alborz 4, were counteracted in Alborz 5 by using a mud of 128–135 lb/cu. ft. in drilling through that section. This time the difficulty of handling a heavy mud, such as the hazards caused by the swabbing effect of the bit while pulling out, was the main difficulty. The Alborz No. 5 well, however, reached the oil horizon, but only after a series of protracted operations of fishing and side-tracking were painstakingly overcome. It may be of interest to note that the side-tracked hole lies so near one of the fish that the drill often touched its end when it was being lowered to the bottom.

At the depth of about 2400–2500 m, gas was occasionally noticed bubbling on the mud surface. The testing of the formation was ruled out on account of extremely precarious conditions existing at the bottom of the hole. No gas bubble, however, was reported immediately before the blow-out.

Thus the drilling of the 8½-inch hole went ahead with 4½-inch drill pipe, 2-5¾-inch × 2½″ i.d. and 10-5-inch o.d. × 2-inch i.d. drill collars. At 2676 m a change of formation was recorded. At about 03·20 on 26 August, the well began to surge and rapidly unload the 129 lb/cu. ft. mud column and it then blew wild. Several streams of oil began flowing away from the wild well. The largest one of these was gauged and was found to be running at about 80,000 b.d. Taking into consideration the oil sprayed around the well, and the oil flowing in smaller streams, it was estimated that the initial oil production amounted to about 100,000 b.d. or even more. This was borne out by the quantity of oil accumulated in several rapidly improvised reservoirs, the construction of which involved about 5 million cu. ft. of earthwork. Some details of these reservoirs are already published.[8]

It should be pointed out that right from the beginning considerable interest was taken by all concerned in evaluating the true reservoir pressure. Thus it was estimated that at the moment the well blew wild the mud column was exerting a pressure of about 8000 p.s.i. at a total depth of about 8782 ft (2677 m). Gas-cutting could not be contemplated because the mud properties were being checked at 15-minute intervals throughout the shift. No change either in the mud properties or in the position of the mud level in the mud pits was observed.

When the capping assembly was installed the ques-

tion of observing the accurate wellhead pressure and thereby estimating the reservoir pressure presented itself.[7] To visualize the situation a brief description of the capping assembly may be helpful. The assembly consisted from bottom to top of:

(1) An adaptor spool which was to be connected to the 7-inch casing spool, to the southern side of which a plug valve had been connected.

(2) A flow spool with 2-4-inch flanged outlets each fitted with two plug valves. When assembly was installed one outlet faced E. and the other faced W.

(3) A hydraulically-operated Cameron D.V. valve.

The western flowline was fitted with a 3-inch positive choke. The eastern flowline was connected through a Shaffer variable choke, having a maximum opening of 1 inch, to a 2000-ft flowline.

A 2-inch W.K.M. gate valve was added to the aforesaid 2-inch plug valve on the 7-inch casing spool in the wellhead assembly facing S. One joint of $2\frac{3}{8}$-inch tubing was screwed into the 2-inch W.K.M. valve. After the above-mentioned $2\frac{3}{8}$-inch tubing was screwed into the W.K.M. valve, both valves were opened, permitting flow through the tubing. The 4-inch plug valves on the E. flow line were opened, permitting flow through the 1-inch choke. The valves on the W. flowline were closed and the wellhead pressure increased immediately to 2500 p.s.i. and remained constant at this value.

An attempt was made to close the W.K.M. valve, but after two turns of the valve wheel the valve could not be closed further, nor could it be reopened. The wellhead pressure had, meanwhile, jumped to 4500 p.s.i. The 4-inch valves were immediately reopened on the W. side and the pressure declined to 2500 p.s.i. A few minutes later the $2\frac{3}{8}$-inch tubing blew out of the W.K.M. valve.

As it was necessary to remove a 4-inch × 2-inch reducer, which had previously been installed on the end of the 4-inch flowline to the W., the 4-inch valves on that side were closed and the pressure built up to 3500 p.s.i. in the few seconds necessary to make the change, and was still increasing when the valves were reopened.

A second attempt to close the 2-inch W.K.M. valve was unsuccessful and the 2-inch valve, fitted before it on the 7-inch casing spool, was necessarily closed. At this time the 1-inch choke on the E. side plugged solidly with debris and cavings from the hole. The well was then flowing to the W. through the 3-inch i.d. positive choke and 4-inch line.

In considering the Alborz reservoir pressure it may be apposite to point out that the salt plug, W. of the over 50-km-long Alborz structure,[5] is believed to be flowing out at a rate sufficient to compensate for the continuous removal of the exposed salt by the seasonal rainwater. The seat of the high pressure required, not only to counterbalance the weight of the Kuh-i-Namak salt plug, but also to induce flow at the said rate, may be the same as that causing the high pressure phenomena observed in the A.5 well.

These facts, taken in conjunction with the stratigraphy[4] and structural geology of the area, leave little doubt as to the extent and the extraordinary condition of the Alborz oil reservoir partially manifested through the A.5 blow-out. The successful capping of the A.5 well was due not only to the selfless devotion of the IOC rank and file, ably led by F. Naficy, Exploration and Production Director of the National Iranian Oil Co., and by B. Mostofi, Managing Director of the Iran Oil Co., but also to an international co-operation, coupled with discipline as well as good fellowship, which has often been declared impossible by short-sighted sectarianism.

ACKNOWLEDGMENTS

The authors wish to thank F. Naficy, NIOC Exploration and Production Director, and also B. Mostofi, IOC Managing Director, for suggesting the preparation of this paper. Liberal assistance given by the IOC engineers and staff is also gratefully acknowledged.

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WELL INTERFERENCE TESTS IN THE SOUTH DOME OF THE ZUBAIR RESERVOIR *

By M. J. E. HEATON †

SUMMARY

Well interference tests were carried out on the three wells in the South Dome of the Zubair Reservoir which have been completed in the Third Pay. For this purpose one of the wells (Well No. Zb 17) was put on stream at 5000 b.d., and minimum closed-in pressure measurements taken at Well No. Zb 2 (3 km away) and Well No. Zb 19 (10 km away) to determine the form of the resultant pressure drawdowns there.

Two studies were carried out. One, after 150 days, concentrated mainly on Well No. Zb 2; and the other after 200 days, when Well No. Zb 19 was reviewed.

The theory of point sinks in an infinite reservoir was applied, using the method of images to allow for the size of the reservoir in the study of Well No. Zb 19. Selected theoretical drawdown curves were obtained. By fitting various such curves to the observed drawdown at Well No. Zb 2 (corrected for overall pressure decline) figures were obtained for the reservoir parameters. These figures may then be fitted into the more complicated equation for Well No. Zb 19.

A check is thus available on the parameters, which have also been determined by laboratory investigation. The main purpose of the tests, however, is to determine the connectivity of the producing sands, *i.e.* what percentage of the sand at one well continues to a point some distance away.

PREVIOUS HISTORY OF THE SOUTH DOME

A rough map of the South Dome, showing the three relevant wells, is appended (Fig 1). It will be seen that the South Dome is an oval structure roughly 20 km long by 10 km broad, with the three wells approximately in a line down the long axis. The average

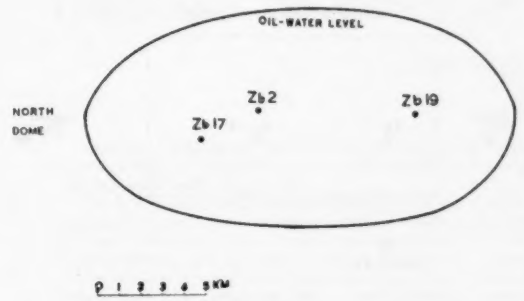

FIG 1

ROUGH MAP OF SOUTH DOME, SHOWING THE THREE WELLS COMPLETED IN THE THIRD PAY

sand thickness above oil–water level is about 120 feet. It is not known whether the structure is faulted off from the North Dome, but certainly there is little if any oil connexion between the two domes. The pay is about 50 per cent sand.

It should be emphasized that these figures are only known very approximately, largely on account of the lack of data from drilled wells.

The total production from the South Dome prior to the commencement of the tests in mid-May was less than 100,000 brl, and there was no production at all for the preceding five months.

A pressure survey was carried out to determine the pressure decline in the dome consequent on the North Dome production. Declines of 0·7 p.s.i/month at Well No. Zb 19, 1·1 p.s.i/month at Well No. Zb 2, and 1·7 p.s.i/month at Well No. Zb 17 were found. Allowance is made for this effect.

GEOLOGICAL EVIDENCE

The Zubair Reservoir consists of a large number of thin sands, separated by silts and shales. Owing to the wide spacing of the wells, particularly in the South Dome, it is impossible to tell with any confidence whether sands at corresponding depths in neighbouring wells are connected or not. A certain amount of negative evidence is available, however, in that certain sands in some wells obviously have no counterpart in their neighbours, or correspond to much thinner sands there.

MATHEMATICAL THEORY

The differential equation for one well in an infinite reservoir is [1]

$$\frac{\partial^2 P}{\partial r^2} + \frac{1}{r}\frac{\partial P}{\partial r} = \frac{\phi \mu c}{k}\frac{\partial P}{\partial t} \quad . \quad . \quad . \quad (1)$$

where P is pressure drawdown in atmosphere at distance r cm from the well at time t sec;

ϕ is porosity, as a fraction;

k is permeability, in darcies;

μ is oil viscosity, in cP;

c is compressibility (of oil and sand combined), in vol/vol/atmosphere.

* MS received 17 June 1957.

† Reservoir Engineer, Basrah Petroleum Co. Ltd.

A solution of equation (1), for a well producing at constant rate q cc/sec (reservoir oil), from sand of thickness h cm, is [2]

$$P = -\frac{\mu q}{4\pi kh} Ei\left(-\frac{\phi\mu c r^2}{4kt}\right) \quad . \quad . \quad (2)$$

where Ei is the exponential integral, defined by

$$-Ei(-k) = \int_k^\infty \frac{e^{-t}}{t}\, dt \quad . \quad . \quad . \quad (3)$$

Solutions of the form (2) may be combined additively. This allows the method of images to be used conveniently to deal with reservoir boundaries, faults, etc.

DISCUSSION

First we must transform equation (2) to a more convenient system of units:

$$P = -\frac{107{\cdot}3\mu q}{kh} Ei\left(-\frac{4{\cdot}26 \times 10^6 \phi\mu c r^2}{kt}\right). \quad (4)$$

where P is pressure drawdown, in p.s.i.;
r is distance from producing well, in km;
t is time of production, in days;
q is production rate, in b.d. (of stock tank oil, assuming a formation volume factor of 1·52);
k is permeability, in mD;
h is formation thickness, in ft;
ϕ is porosity, as a percentage;
μ is viscosity, in cP;
c is compressibility, in vol/vol/p.s.i.

Note that h is average connected sand thickness between the two wells in question.

Fig 2 shows the observed drawdown at Well No. Zb 2 after 150 days, corrected for overall pressure decline, compared with the curve in

$$P = -21Ei\left(-\frac{27}{t}\right). \quad . \quad . \quad . \quad (5)$$

which is seen to provide a good fit.

Remembering that $r = 3$ for Well No. Zb 2, we have

$$3 = 4{\cdot}26 \times 10^6 \times \frac{\mu}{k} \times \phi c;$$

$$21 = 107{\cdot}3 \times \frac{\mu}{k} \times \frac{q}{h} \quad . \quad (6)$$

Now laboratory investigations give fairly accurate measurements of f and c as 19 per cent and 16×10^{-6} vol/vol/p.s.i. respectively. Also $q = 5000$. Hence we have

$$\frac{\mu}{k} = 0{\cdot}00232; \quad h = 59{\cdot}3. \quad . \quad . \quad (7)$$

Using the laboratory figure $\mu = 0{\cdot}6$, this gives $k = 259$, which agrees, within the limits of error, with laboratory measurements.

Turning attention now to well No. Zb 19, and assuming that the parameters deduced above remain constant (with the exception of the connected sand thickness h) we may adopt the general equation

$$P = -\frac{1250}{h} Ei\left(-\frac{3r^2}{t}\right) \quad . \quad . \quad (8)$$

as the pressure drawdown due to a given well.

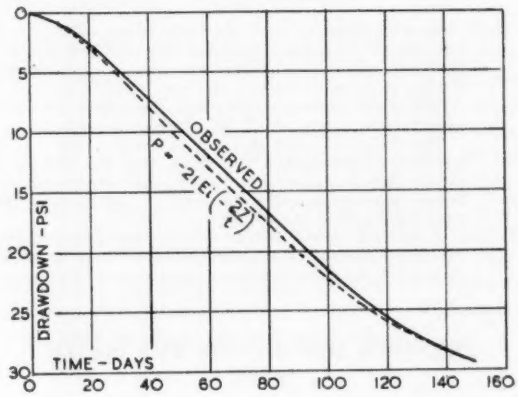

Fig 2

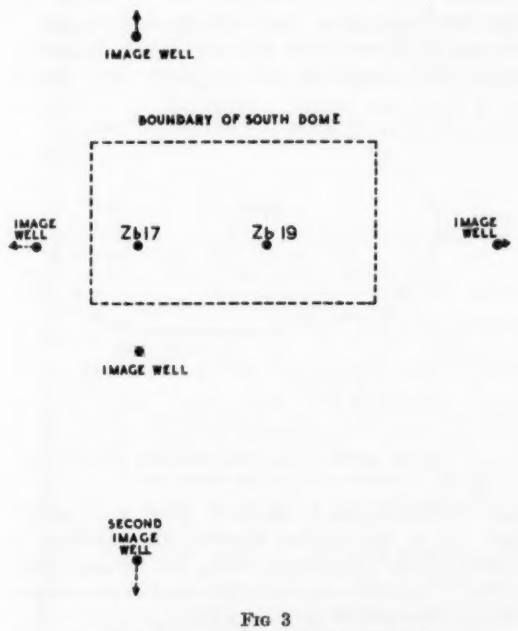

Fig 3
IMAGE REPRESENTATION OF SOUTH DOME

The assumption that the parameters other than h remain constant will be justified if a good fit is obtained. As will be seen, any variation in them must be small compared to the variation in h.

From Fig 1, it is clear that one must take into

account the size of the reservoir when dealing with wells as far apart as Wells No. Zb 17 and Zb 19. This is done by the method of images; the field is represented by a series of wells in an infinite reservoir, as in Fig 3.

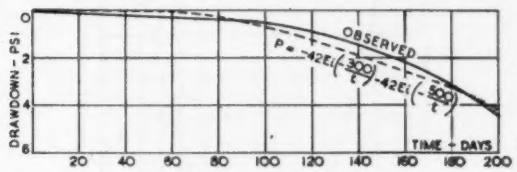

FIG 4

In practice, it is found sufficient just to use the one "image," at 13 km from Well No. Zb 19, since for each of the others the resultant drawdown after 200 days is of the order of 0·01 p.s.i. or less. This gives

$$P = -\frac{1250}{h} Ei\left(-\frac{300}{t}\right) - \frac{1250}{h} Ei\left(-\frac{500}{t}\right). \quad (9)$$

for the pressure drawdown of Well No. Zb 19 consequent on the production at Well No. Zb 17.

Fig 4 shows the graph of equation (9), with $h = 30{\cdot}5$ compared with the observed drawdown corrected for overall pressure decline. Again a good fit is obtained.

CONCLUSION

The theoretical investigation of well interference tests herein carried out gives results in good agreement with laboratory investigations, and also sheds light on the connectivity of the sands in the South Dome.

It is suggested that about half the sand thickness in the individual wells connects between Wells No. Zb 17 and Zb 2, a distance of about 3 km; and about a quarter between Wells No. Zb 17 and Zb 19, a distance of about 10 km.

ACKNOWLEDGMENT

The author wishes to thank the Basrah Petroleum Co. Ltd, for permission to publish this paper.

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DISTRIBUTION OF FLUID PROPERTIES IN A HIGH RELIEF OILFIELD AND ITS EFFECT UPON THE MATERIAL BALANCE*

By H. K. GRANT†

SUMMARY

Vertical variation in the fluid properties of high relief oil reservoirs has been recognized for several years, but there is little or no reference in the literature on its significance in estimating the tank oil originally in place by the material balance method.

Using a hypothetical reservoir saturated at or near the crest with the variation in the fluid properties with elevation based upon published data, it was found that no single p–v–t can be used in calculating the oil in place by material balances. It was found that the reservoir must be numerically integrated by dividing it into horizontal layers, each having its own p–v–t characteristics.

Curves are presented to show the probable variation with elevation of the fluid properties of a hypothetical reservoir. Also presented are material balance calculations based upon three methods: (1) using a variable p–v–t (numerical integration method); (2) using a volumetrically weighted p–v–t; and (3) using a p–v–t based upon the average tank oil produced.

INTRODUCTION

THE fact that the physical properties of fluids vary with elevation in a single oil reservoir has been recognized for many years. Sage and Lacey in 1939 did some theoretical work predicting changes in the physical properties with structural position.[1] Since then several authors have noted increasing fluid densities with depth, particularly on high relief pools.

The purpose of this paper is two-fold: first, to show, using a hypothetical reservoir, how the fluid properties might vary with elevation based upon published data; secondly, to calculate the effect these variations would have in a material balance type of reserve estimate using a volumetrically weighted p–v–t and one based upon the average produced oil as compared to a more realistic consideration of the variation in the p–v–t with elevation.

It can safely be said that almost all of the work done to date on small reservoirs involved the use of a single p–v–t. For most of the cases the errors in using a single or average p–v–t in estimating oil and

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gas reserves by material balances are probably small, but for some the errors could be quite substantial and may explain, at least in part, why many material balance calculations have given erroneous results.

The hypothetical reservoir used in showing the variation in the physical properties of the fluids and the importance of considering the reservoir in horizontal layers relative to p–v–t data is one having a total relief of 1000 ft. Several curves are presented showing the variation in the physical properties with elevation and pressure. All p–v–t data were determined by applying the physical properties to Standing's empirical p–v–t correlations for mixtures of California oils and gases.[2] Also presented are example calculations for three material balances based upon: (1) a variable p–v–t; (2) a volumetrically weighted p–v–t; and (3) a p–v–t based upon the average produced oil.

DISTRIBUTION OF FLUID PROPERTIES

The amount of published data on the estimation of oil and gas reserves by material balances and the prediction of the future performance of oil reservoirs by considering the variation in the physical properties of the contained fluids with structural position is extremely limited. Probably the first complete reservoir analysis which considered a changing p–v–t with elevation is the one presented by D. R. McCord [3] on the performance predictions of the LL-370 area, Bolivar Coastal field, Venezuela. It is shown in his paper that the oil gravity varied between 18° and 28° API over 1500 ft of closure for an average gravity change of about 0·7° API per 100 ft of elevation. In the fifth zone of the Newhall-Potrero field in California there occurred a variation of tank oil gravity from 25° API to 39° API within a closure of 2000 ft.[4] The average change in API gravity over the range of closure in this field was 0·7° API per 100 ft, which agrees with the variation found in the Bolivar Coastal field. The initial saturation pressure in the Weber sand of the Rangely field in Colorado varied from 2560 to 1960 p.s.i. over 840 ft of relief [5] for an average saturation pressure change of about 71 p.s.i. per 100 ft of elevation. Similar variations have been found in the large Burgan field in Kuwait.

In developing a hypothetical reservoir for the purpose of illustrating the variation of the physical properties and in showing the effect of these variations on material balance calculations, an average change of 0·7°API per 100 ft and an average change of 70 p.s.i. per 100 ft of elevation in the initial saturation pressure was used to agree in general with known data on high relief pools. The variation in the tank oil gravity and initial saturation pressure with elevation should not be taken as linear. An oilfield with a closure of 1000 ft between the elevations of 6000 and 7000 ft is assumed. A circular homogeneous reservoir with a 5-mile radius containing a constant net sand thickness of 200 ft, a porosity of 28 per cent, and water saturation of 10 per cent of the pore volume is assumed. Other values for the porosity and water

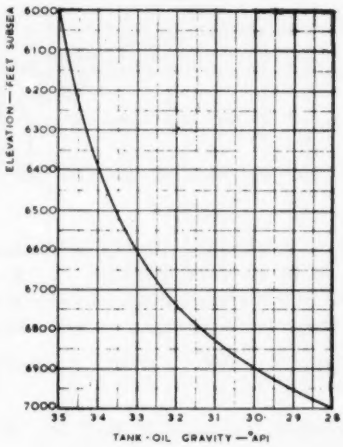

FIG 1

TANK OIL GRAVITY v. ELEVATION

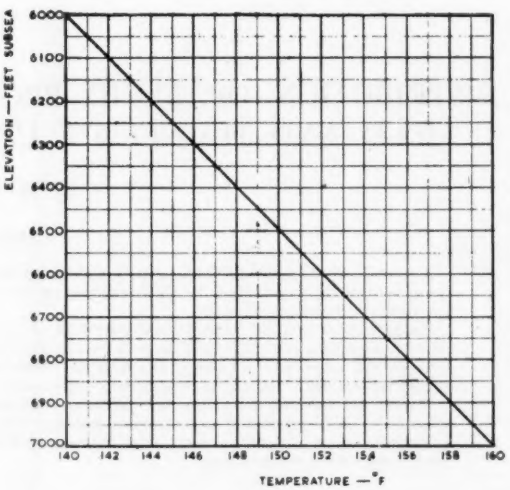

FIG 2

TEMPERATURE v. ELEVATION

saturation could have been used without affecting the conclusions at the end of the paper.

The change in the tank oil gravity is assumed to vary between 28° API and 35° API (Fig 1); the reservoir temperature to vary between 140° and 160° F (Fig 2). The initial solution ratio was assumed to range from 400 to 700 cu. ft. per barrel of tank oil (Fig 3). The gravity of the produced gas was assumed to vary between 0·77 and 0·83 (Fig 4), and the gravity of the free gas was taken as 0·70 over the reservoir

conditions used in the material balance calculations. Fig 5 shows the original saturation pressure–elevation relationship for the assumed reservoir, obtained by

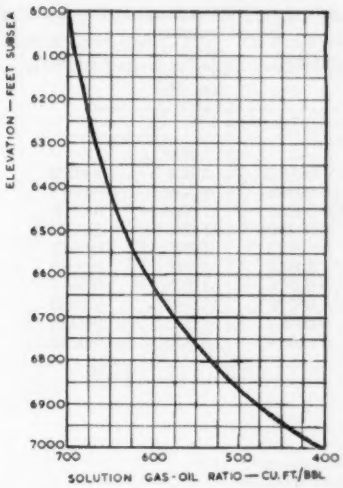

FIG 3

GAS SOLUBILITY *v.* ELEVATION

applying the physical properties of the fluids in Standing's empirical *p–v–t* correlations for mixtures of California oils and gases. The empirical *p–v–t* relationships established by Standing were obtained by running numerous *p–v–t* on various types of California crudes. The relationships are normally presented in the form of three charts whereby the saturation pressure and liquid phase formation volume factor can be obtained by knowing such field data as the producing gas–oil ratio of a bubble point liquid, and the produced oil and gas gravities plus the reservoir temperature. The variation in the original pressure checks the 70 p.s.i. per 100 ft of elevation observed in actual reservoirs. In developing the original reservoir pressure–elevation relationships also shown on Fig 5, it was assumed that the reservoir was just saturated at the top.

The original liquid phase formation volume factor was obtained by applying the liquid phase compressibility obtained from Fig 6 showing the compressibility above the saturation pressure as a function of the saturation pressure. This curve was

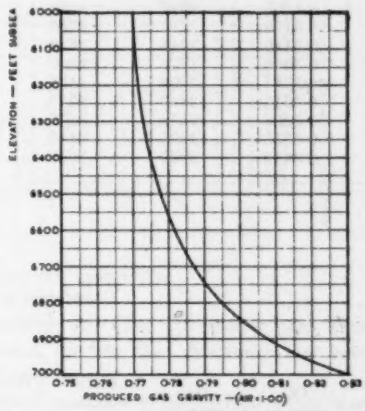

FIG 4

PRODUCED GAS GRAVITY *v.* ELEVATION

obtained by drawing a smooth curve through some thirty measurements of saturation pressure and liquid phase compressibility. It is recognized that this curve is only roughly correct and also that the liquid phase compressibility above the saturation pressure is not constant but decreases slightly with increasing pressure. However, this error can be considered negligible. Table I shows the calculation of the

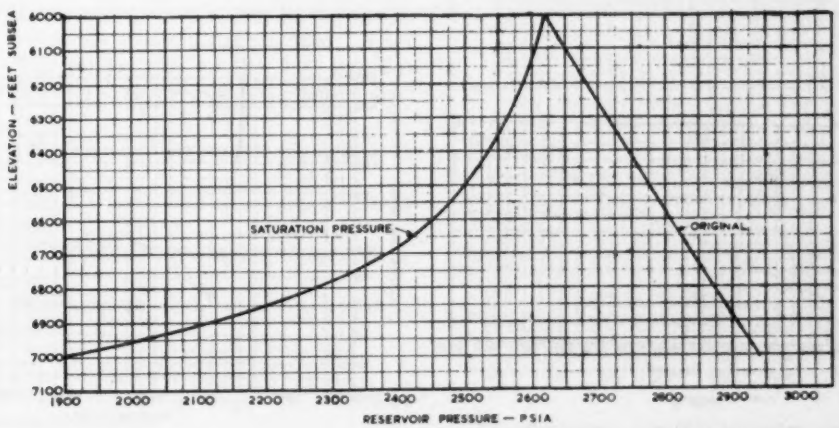

FIG 5

ORIGINAL AND SATURATION PRESSURE *v.* ELEVATION

TABLE I

Calculation of Original Liquid Phase Formation Volume Factor

Elevation, ft subsea	Gradient, p.s.i/ft	Reservoir pressure, P, p.s.i.a.	Saturation pressure, P_s, p.s.i.a.	ΔP	Compressibility, C, per 10^6 p.s.i.	$C\Delta P$	Formation volume factor at P_s, B_{os}, Res. Brl/ Brl T.O.	$C\Delta PB_{os}$	Original formation volume factor, $B_{oi} = B_{os} - C\Delta PB_{os}$
6000	0·3081	2620	2620	—	13·80	—	1·360	—	1·360
6100	0·3095	2651	2605	46	13·73	0·00063	1·354	0·0009	1·353
6200	0·3105	2682	2590	92	13·65	0·00126	1·350	0·0017	1·348
6300	0·3120	2713	2565	148	13·53	0·00200	1·343	0·0027	1·340
6400	0·3140	2744	2535	209	13·39	0·00280	1·335	0·0037	1·331
6500	0·3160	2776	2500	276	13·22	0·00365	1·327	0·0048	1·322
6600	0·3181	2808	2455	353	13·00	0·00459	1·318	0·0060	1·312
6700	0·3213	2840	2385	455	12·66	0·00576	1·305	0·0075	1·298
6800	0·3256	2872	2275	597	12·15	0·00725	1·288	0·0093	1·279
6900	0·3323	2905	2110	795	11·32	0·00900	1·262	0·0114	1·251
7000	0·3412	2939	1900	1039	10·30	0·01070	1·230	0·0132	1·217

TABLE II

Calculation of Original Reservoir Pressure Gradient

Elevation, ft subsea	1 Oil specific gravity, ρ_o	2 Gas gravity air = 1·00, ρ_g	3 Initial solution ratio, R_s, cu. ft/brl	4 Wt of tank oil per brl, $350{\cdot}16 \times \rho_o$, lb	5 Wt of produced gas per cu. ft., $0{\cdot}076362 \times \rho_g$, lb	6 Wt of gas per brl of tank oil, $0{\cdot}076362 \times \rho_o \times R_s$, lb	7 Wt of oil plus gas per brl T.O., 4 + 6, lb	8 Original liquid phase formation volume factor, B_{oi}, brl/brl	9 Initial reservoir pressure gradient, $\frac{7 \times 0{\cdot}4331}{B_i \times 350{\cdot}16}$, p.s.i/ft
6000	0·8498	0·770	700	297·57	0·0588	41·14	338·73	1·360	0·3081
6100	0·8509	0·771	690	297·95	0·0589	40·64	338·59	1·353	0·3095
6200	0·8519	0·772	680	298·30	0·0589	40·05	338·35	1·348	0·3105
6300	0·8534	0·773	665	298·83	0·0590	39·24	338·07	1·340	0·3120
6400	0·8550	0·775	650	299·39	0·0592	38·48	337·87	1·331	0·3140
6500	0·8576	0·778	630	300·30	0·0594	37·42	337·72	1·322	0·3160
6600	0·8602	0·782	607	301·21	0·0597	36·24	337·45	1·312	0·3181
6700	0·8639	0·787	576	302·50	0·0601	34·62	337·12	1·298	0·3213
6800	0·8686	0·795	535	304·15	0·0607	32·47	336·62	1·279	0·3256
6900	0·8762	0·808	475	306·81	0·0617	29·31	336·12	1·251	0·3323
7000	0·8871	0·830	400	310·63	0·0634	25·36	335·99	1·218	0·3412

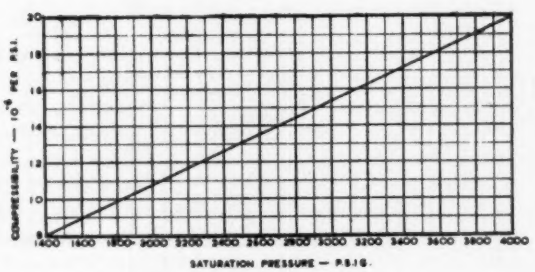

FIG 6

SATURATION PRESSURE *v.* LIQUID COMPRESSIBILITY

Compressibility of well fluids above the saturation pressure as a function of saturation pressure.

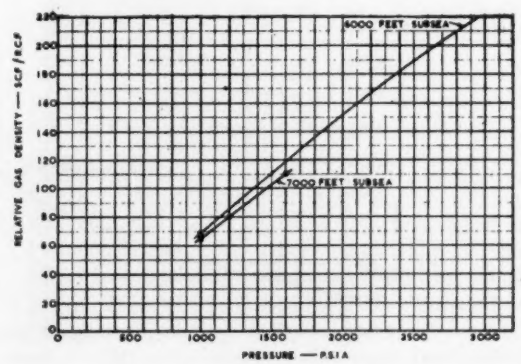

FIG 7

RELATIVE GAS DENSITY *v.* PRESSURE

original liquid phase volume factor. The original reservoir pressure gradient was checked by calculating the density of the liquid and gas phases. This calculation is shown on Table II.

The relative gas density for the free gas was obtained by assuming a free gas gravity of 0·70 at reservoir conditions over the pressure range used in the material balance calculations and determining the compressibility of the free gas from the gas gravity using the relationship that the pseudo critical pressures and temperatures are functions of the gas gravity. This method is explained by G. G. Brown.[6] Fig 7 shows the relative gas density as a function of pressure. Figs 10 and 11 are the working curves for the gas solubility and liquid phase formation volume factor obtained from the given data using Standing's p–v–t correlations.

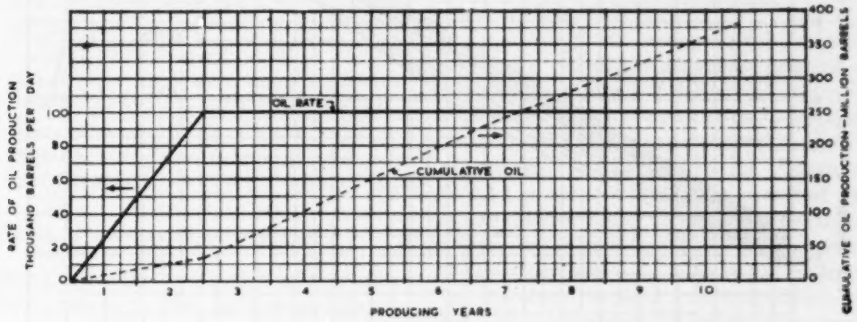

FIG 8

POOL PERFORMANCE OF A GIVEN HYPOTHETICAL RESERVOIR

ASSUMPTIONS

The past performance of the hypothetical reservoir and the material balance calculations are based on the following assumptions:

(1) The reservoir consists of four layers, within each of which the properties of the fluid are the same. This applies only to the first material balance case. The layers and mean depths used are—

6000–6400 ft subsea with mean depth 6300 ft subsea.
6400–6600 ft subsea with mean depth 6500 ft subsea.
6600–6800 ft subsea with mean depth 6700 ft subsea.
6800–7000 ft subsea with mean depth 6900 ft subsea.

(2) Little or no gravity drainage.

(3) Constant free gas gravity over the range of pressures considered.

(4) Tank oil and produced gas gravities do not change with time at a given elevation.

(5) Reservoir initially saturated at the crest.

(6) All oil is produced between the elevations of 6400 and 6800 ft subsea. Although this assumption is valid for material balance case No. 1, slight changes in the location of the average p–v–t in the upper and lower zones would occur, due to drainage into the middle zones. However, for this particular case this can be ignored due to the relatively low rates of oil withdrawal.

(7) Expansion of interstitial water is negligible.

(8) No form of pressure maintenance.

(9) No effect from rock compressibility.

(10) No water drive.

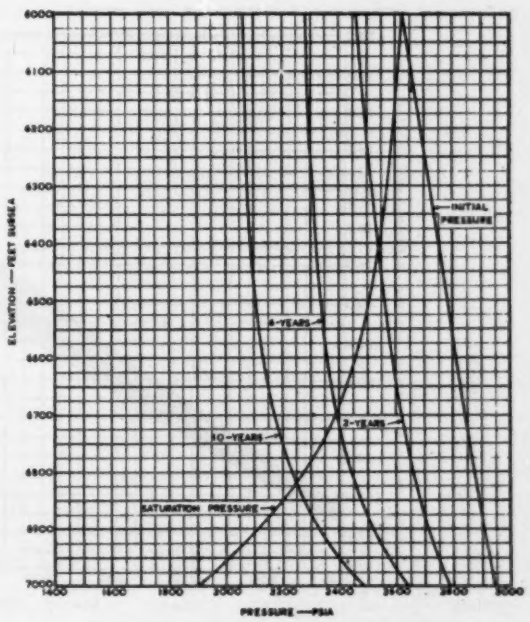

FIG 9

RESERVOIR AND SATURATION PRESSURES v. ELEVATION
Production periods of 2, 6, and 10 years.

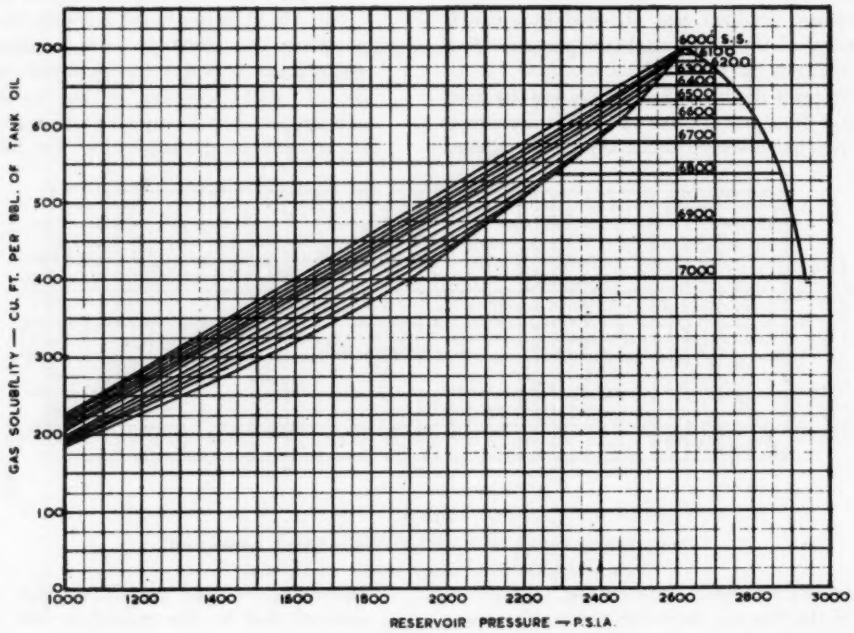

FIG 10

GAS SOLUBILITY *v.* RESERVOIR PRESSURE

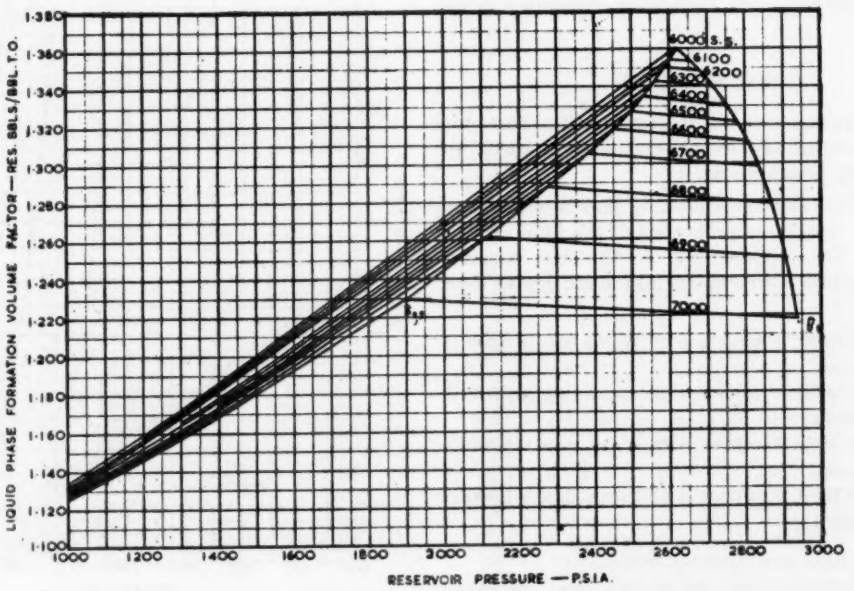

FIG 11

LIQUID PHASE FORMATION VOLUME FACTOR *v.* RESERVOIR PRESSURE

MATERIAL BALANCE BY LAYERS

A standard form of the material balance may be written as follows:[7]

$$N = \frac{N_c(\gamma B_o - R) + G_c}{(R_i - R) - \gamma(B_{oi} - B_o)}$$

where N = oil originally in place in brl;
N_c = cumulative oil produced in brl = ΣNcn;
γ = relative gas density × 5·615 cu. ft/brl in S.C.F. per reservoir barrel;
B_o = liquid phase formation volume factor in reservoir barrels per brl of tank oil;
G_c = cumulative gas production in cu. ft.;
B_{oi} = initial liquid phase volume factor;
R = solution gas–oil ratio in cu. ft/brl of tank oil;
R_i = initial solution gas–oil ratio.

When more than one layer is used in a reservoir to consider the variation in the fluid properties the material balance equation can be written:

$$N = \frac{\Sigma[N_{cn}(\gamma B_o - R)] + G_c}{\Sigma\{f_n[(R_i - R) - \gamma(B_{oi} - B_o)]\}}$$

where f denotes the ratio of the tank oil originally in place in each layer to the total tank oil originally in place in the reservoir. This equation requires the introduction of the relative value of the tank oil in place from the volumetric estimate and a knowledge of oil production from each layer.

In showing the comparison of the results obtained between p–v–t data that varies with elevation, a volumetric average p–v–t and one based on the average produced oil, it is assumed that the tank oil originally in place was 12·3 × 10⁹ brl to agree with the volumetric estimate. It should be emphasized, however, that other values for the tank oil originally in place could be used without affecting the conclusions. In other words, the volumetric estimate was made only for the purpose of obtaining the f factor used in the material balance equation.

Fig 8 shows the assumed pool performance curve and Fig 9 the assumed average pressure–depth relationship at production periods of 2, 6, and 10 years. The average rate of production is assumed at about 100,000 b.d. The pressure–depth curves used were arbitrarily made to give values of tank oil in place of 12·3 × 10⁹ brl, using the material balance case where the p–v–t data varies with elevation. In other words, recognizing that this type of material balance is the most realistic. The other material balances are calculated to show how inconsistent their results are and how greatly their answers vary from the assumed realistic answer.

Table III shows a material balance calculation based upon a producing period of ten years, using the method of dividing the reservoir into four separate layers. From the calculations it is clearly evident the most critical factor in the material balance is the saturation pressure–elevation relationship which determines the value of $f(R_i - R)$. Because of the critical nature of this factor, the reservoir is separated into small layers, each 100 ft in thickness, in the region where the pressures have fallen below the initial saturation pressures.

TABLE III

Material Balance on a Given Hypothetical Reservoir after a Producing Period of Ten Years using a Changing p–v–t

$N = \frac{\Sigma[N_c(\gamma B_o - R)] + G_c}{\Sigma\{f[(R_i - R) - \gamma(B_{oi} - B_o)]\}}$	Symbol	Unit	Top to 6400 ft s.s., 6300 ft p–v–t, f = 0·1098	6400–6600 ft s.s., 6500 ft p–v–t, f = 0·1915	6600–6800 ft s.s., 6700 ft p–v–t, f = 0·2917	6800–7000 ft s.s., 6900 ft p–v–t, f = 0·4070	Total
Cumulative production:							
Oil	N_c	Brl × 10⁶	—	190	190	—	—
Gas	G_c	M.c.f. × 10⁶	—	129	129	—	—
Pressure	P	P.s.i.a.	2078	2105	2172	2322	—
Relative gas density	γ	S.C.F/R.C.F.	157	158	163	166	—
Liquid phase $f.v.f.$	B_o	Res. brl/brl T.O.	1·275	1·272	1·276	1·258	—
γB_o		—	—	201	208	—	—
$5{\cdot}615\gamma B_o$		—	—	1129	1168	—	—
Solution gas–oil ratio	R	S.C.F/brl T.O.	—	510	507	—	—
$5{\cdot}615\gamma B_o - R$		S.C.F/brl T.O.	—	619	661	—	—
$N_c(5{\cdot}615\gamma B_o - R)/1000$		M.c.f. × 10⁶	—	118	126	—	—
$N_c(5{\cdot}615\gamma B_o - R)/1000 + G_c$		M.c.f. × 10⁶	—	247	255	—	502
Initial liquid phase $f.v.f.$	B_{oi}	Res. brl/brl T.O.	1·340	1·322	1·298	1·251	—
$(B_{oi} - B_o)$		Res. brl/brl T.O.	+ 0·065	+ 0·050	+ 0·022	−0·007	—
$\gamma(B_{oi} - B_o)$		—	+10·205	+ 7·900	+ 3·586	−1·162	—
$5{\cdot}516\gamma(B_{oi} - B_o)$		S.C.F/brl T.O.	+57·301	+44·359	+20·135	−6·525	—
$-f[5{\cdot}615\gamma(B_{oi} - B_o)]$		S.C.F/brl T.O.	− 6·292	− 8·495	− 5·873	+2·656	−18·004
$\Sigma f(R_i - R)$		S.C.F/brl T.O.	16·624	23·416	18·612	—	58·652
							40·648
Tank oil in place	T.O.I.P.	Brl × 10⁹	—	—	—	—	12·34

MATERIAL BALANCE USING WEIGHTED AVERAGE *p–v–t*

The weighted average *p–v–t* was based upon the mean gravity of the tank oil originally in place. The mean elevation of this average oil is taken from Fig 12

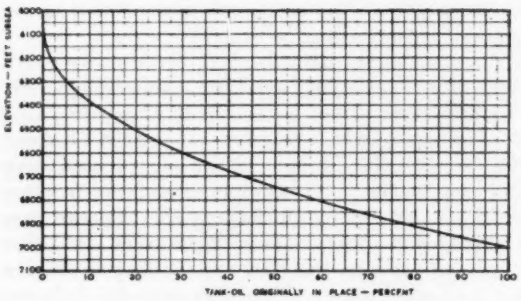

FIG 12

PER CENT TANK OIL ORIGINALLY IN PLACE *v.* ELEVATION

For a homogeneous circular reservoir having a 5-mile radius with constant dip and uniform net productive sand thickness of 200 ft.

at about 6750 ft subsea. A material balance after ten years of production using the *p–v–t* at this elevation is shown in Table IV. In analysing the calculations it is clearly evident that this type of *p–v–t* does not consider the average effect of the reservoir pressure on the solution ratio but only the effect at one elevation. Although this material balance gave a value of tank oil in place of $14{\cdot}8 \times 10^9$ brl with an error of only 16·9 per cent, the material balance after six years of production using this *p–v–t* resulted in a tank oil in place of $52{\cdot}1 \times 10^9$ brl with an error of over 300 per cent.

TABLE IV

Material Balance on a Given Hypothetical Reservoir after a Producing Period of Ten Years

A = using a weighted average *p–v–t*
B = using an average tank oil *p–v–t*

$N = \frac{N_p(\gamma B_o - R) + G_p}{(R_1 - R) - \gamma(B_{oi} - B_o)}$	Symbol	Unit	A 6750 ft *p–v–t*	B 6600 ft *p–v–t*
Cumulative production:				
Oil	N_p	Brl × 10^9	380	380
Gas	G_p	M.c.f. × 10^9	258	258
Pressure	P	p.s.i.a.	2300	2132
Relative gas density	γ	S.C.F/R.C.F.	165	161
Liquid phase *f.v.p.*	B_o	Res. brl/brl T.O.	1·279	1·274
γB_o		—	211·035	205·110
$5{\cdot}615\gamma B_o$		—	1185	1152
Solution gas–oil ratio	R	S.C.F/brl T.O.	512	507
$5{\cdot}615\gamma B_o - R$		S.C.F/brl T.O.	673	645
$N_p(5{\cdot}615\gamma B_o - R)/1000$		M.c.f. × 10^9	256	245
$N_p(5{\cdot}615\gamma B_o - R)/1000 + G_p$		M.c.f. × 10^9	514	504
Initial liquid phase *f.v.f.*	B_{oi}	Res. brl/brl T.O.	1·288	1·312
$(B_{oi} - B_o)$		Res. brl/brl T.O.	+0·009	+0·038
$\gamma(B_{oi} - B_o)$		—	+1·485	+6·118
$-5{\cdot}615\gamma(B_{oi} - B_o)$		S.C.F/brl T.O.	−8·337	−34·353
$(R_1 - R)$		—	43	123
Tank oil in place	T.O.I.P.	Brl × 10^9	14·83	5·68

MATERIAL BALANCE USING A *p–v–t* AT AN ELEVATION OF 6600 FT SUBSEA

A material balance based upon the average elevation of produced oil after the ten-year production period is shown in Table IV. The calculated tank oil in place in this case was only $5{\cdot}7 \times 10^9$ brl or 54 per cent lower than the given value of $12{\cdot}3 \times 10^9$ brl. In this case too much weight was placed on the change in the solution ratio at this elevation and time relative to the average change in the reservoir.

SUMMARY AND CONCLUSIONS

Table V shows a summary of the results of the three material balance methods at two , six-, and ten-year periods and how they differ from the given value of the tank oil in place.

TABLE V

Summary of Material Balance Results

	Method		
	Changing *p–v–t*	Weighted average *p–v–t*	Average tank oil *p–v–t*
Assumed tank oil originally in place, billion brl	12·3	12·3	12·3
Calculated tank oil in place (two years' production)	12·3	16·6	18·4
Error, %	—	+ 35·0	+49·6
Calculated tank oil in place (six years' production)	12·3	52·1	6·6
Error, %	—	+323·6	−46·3
Calculated tank oil in place (ten years' production)	12·3	14·8	5·7
Error, %	—	+ 16·9	−53·7

Separating the reservoir into layers for the purpose of a material balance becomes even more important when an initial gas cap is present.

Partial water drive could have been assumed for the hypothetical reservoir, but the comparative results would have been quite similar to those obtained. In a given highly undersaturated reservoir it becomes evident that the results of a material balance is dependent entirely on the compressibility of the liquid phase, and therefore the proper *p–v–t* would be one based upon the produced oil as long as the reservoir is everywhere above the saturation pressure.

The following are the important conclusions that can be derived from this paper:

(1) The most realistic method of determining the value of the tank oil in place by the material balance in an oil reservoir that is saturated at or near the crest is one that considers the variation in the fluid properties with elevation. This is clearly evident from the inconsistency of the other two material balance results.

(2) Where the fluid properties vary with elevation in a reservoir saturated at or near the crest, the saturation pressure–elevation relationship is the most critical factor in estimating the reserves by the material balance method.

ACKNOWLEDGMENTS

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APPENDIX

The following development is to show that there is very little error in the material balance results due to oil migration by assuming that all of the production is obtained from the interval between the elevations 6400 ft subsea and 6800 ft subsea.

The gas balance for the assumed production block may be written as:

$$NR_i = (N-N_e)R - N_mR_m + [NB_i - (N-N_e)B - N_mB_m]\gamma + G_e \quad (1)$$

or:

$$NR_i + N_m(R_m - \gamma B_m) = (N-N_e)R + [NB_i - (N-N_e)B]\gamma + G_e \quad (2)$$

where: N = oil originally in place;
R_i = original solution gas–oil ratio;
N_e = cumulative tank oil;
R = solution gas–oil ratio;
N_m = migrated oil;
R_m = solution gas–oil ratio for migrated oil;
B_i = initial liquid phase formation volume factor;
B = formation volume factor;
B_m = formation volume factor of migrated oil;
γ = relative gas density for the free gas in the reservoir;
G_e = cumulative gas produced.

The maximum error in assuming that the migrated oil term $N_m(R_m - \gamma B_m)$ is equal to $N_m(R - \gamma B)$ can be obtained by comparing the maximum and minimum probable values for N_m, R_m, and B_m.

It can be assumed that in calculating the oil reserves by the material balance the maximum probable oil that will be drained into the producing block would not exceed 25 per cent of the tank oil originally in place in the reservoir when the cumulative produced tank oil is about 50 per cent of the oil originally in place.

Further the maximum probable difference between $N_m(R_m - \gamma B_m)$ and $N_m(R - \gamma B)$ for the reservoir conditions used in the report is obtained by assuming that the migrated oil is all from below. This condition, however, can never be attained because there will always be some oil migration into the producing zone from above. The maximum possible value for $(R_m - \gamma B_m) - (R - \gamma B)$ will not exceed 22 per cent of R_i.

Therefore the maximum error introduced by neglecting the second term on the left side of equation (2) is $0{\cdot}25 \times 22 = 5{\cdot}5$ per cent. This is also the error in the calculated value of N, the tank oil in place.

In general the material balance will only be applied when the cumulative production N_e is very much less than 50 per cent of N and N_m is proportionally small. Moreover, part of the drainage will be up and part down so that the difference in the values of $\gamma B - R$ will be less. Hence the error introduced by neglecting the term for the migrated oil will be far less than 5·5 per cent.

ANALYSIS OF PRESSURE DECLINE *

By H. SAMARA †

SUMMARY

A well-known principle, the principle of superposition of states, is used as a basis for a new approach to the analysis of pressure decline curves. This principle is used explicitly or implicitly by every analysis of variable production. In the present investigation no other principle is used explicitly, and the analysis is valid for any laws of flow and for any geometrical boundaries of the reservoir, as long as the principle of superposition remains true.

The analysis enables the prediction of: (1) the bottom hole pressure differential from short flow in wells; (2) future pressure decline in the reservoir, provided that the reservoir has been produced long enough.

EXPERIMENTAL PROCEDURE

(1) Bottom hole pressure differentials are usually measured in the following way. The well is left closed-in long enough for a static bottom hole pressure to be established, the pressure is measured, the well is allowed to flow at a constant rate until the bottom hole pressure stabilizes, then the pressure is measured, the well closed in, and, after the pressure stabilizes, the bottom hole pressure is measured again to ensure its return to its original pressure. The difference between the static and flowing pressures is the bottom hole differential pressure for that rate of flow.

The only drawbacks to this procedure are:

(*a*) the time of flow for stabilizing bottom hole pressure may be unduly long, and this is especially inconvenient for a new well;

(*b*) since the flowing bottom hole equilibrium pressure is not known, the only criterion for judging whether equilibrium has been reached is the rate of change of pressure with time. In certain cases this may be too small to notice long before equilibrium pressure is reached.

(2) In the producing life of a field, pressure decline is measured periodically in observation wells, and a record is kept of production.

A plot of pressure drop *v.* time shows not one smooth monotonic curve which could be easily analysed, but a curve with ups and downs due to changes of production rates. A pressure decline *v.* production rate reflects changes, but to a lesser degree. These changes make the prediction of the pressure behaviour of the reservoir by graphical extrapolation rather uncertain. The usual procedure is to postulate a physical model of the reservoir, work out the behaviour of the reservoir for constant production, and deduce from that the behaviour for the prescribed production and, finally, to match the pressure history with the observed one. When a good match is obtained, an extrapolation is carried out into the future based on the physical model.

The reliability of the prediction depends to a large extent on the length of the record of observation.

The method proposed here would enable one to produce a pure pressure decline curve, *i.e.* a curve for constant production rate, from an actually observed curve with varying production rates. If the reservoir has been produced for a period long enough to obtain the full pure pressure decline curve, *i.e.* for the initial transient pressure drop to have died away, then this curve can be used directly for the purposes of pressure decline prediction, thus eliminating the necessity of introducing a physical model. Even when such a model is required, it is easier to deduce it from the pure curve than from the observed curves.

THEORETICAL ANALYSIS

Principle of Superposition of States

The principle states that the effect of production on pressure decline is additive, *i.e.* that the pressure decline at any given time due to a production rate which is the sum of several production rates is the sum for the same given time of the individual pressure declines due to each production rate separately. The mathematical expression of the principle is as follows.

If, when a reservoir is produced at any rate $q_1(t)$ its pressure decline at time t is $F_1(t)$, and when it is produced at rate $q_2(t)$ its pressure decline is $F_2(t)$, then when it is produced at production rate $q_1(t) + q_2(t)$ its pressure decline would be $F_1(t) + F_2(t)$.

(1) This principle depends on the linearity of the equations of motion, which is the case for the Darcy single fluid flow equations. However, if the equations of motions are non-linear, they can usually be replaced for a first approximation by linear equations, and the smaller the rates of flow under consideration the better the approximation.

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(2) It also depends on the fact that the conditions of the reservoir, like permeability, porosity, and its physical boundary conditions, are unaffected by the flow.

(3) The principle does not depend on the geometrical boundaries of the reservoir, or on the distribution of porosity or permeability inside it.

(4) The principle implies that the B.H.P.D. is proportional to production rate, which is not true in fractured limestone fields in the Middle East.

Nevertheless, the principle is implicitly used whenever a model of the reservoir is employed to deduce pressure drop for variable production rates.

(5) Let the pressure decline for unit constant production rate be $f(t)$, where t is the time since production commenced, then the pressure decline for constant production rate q is $qf(t)$. If production started at $t = T$, and remained constant at the rate q, then the pressure decline is zero from $t = 0$ to $t = T$ (since there has been no production in this period) and is $qf(t - T)$ for $t > T$. Or since $f(t) = 0$ for $t \leqslant 0$, the pressure decline for this case is $qf(t - T)$.

The function

$$D = q[(f(t - T_1) - f(t - T_2)]. \quad . \quad (1)$$

for T_2 more than T_1 represents the pressure decline due to a constant production rate q from time T_1 to T_2. This is so because the first term represents the pressure decline due to production rate q beginning at time T_1, and the second term represents the effect of production $-q$ beginning at time T_2; and hence the combination represents the effect of production q from T_1 to T_2. It is, of course, zero for t less than T_1.

(6) The above result can be generalized for the step function of production

$$\begin{aligned} &q_1 \text{ from } 0 \text{ to } t_1 \\ &q_2 \text{ from } t_1 \text{ to } t_2 \\ &\quad . \quad . \quad . \quad . \quad . \quad . \quad . \\ &q_n \text{ from } t_{n-1} \text{ to } t_n \end{aligned}$$

to give the pressure decline

$$\begin{aligned} D = {} & q_1[(f(t) - f(t - t_1)] + q_2[(f(t - t_1) - f(t - t_2)] \\ & + q_3[(f(t - t_2) - f(t - t_3)] + \quad . \quad . \quad . \quad . \quad . \quad . \\ & + q_n[(f(t - t_{n-1}) - f(t - t_n)] \quad . \quad . \quad . \quad . \quad (2) \end{aligned}$$

(7) For the case of a continuous production rate $q(t)$, the above equation becomes

$$D = \int_0^t q(u)\, f'(t - u)\, du \quad . \quad . \quad (3)$$

(8) The pure curve, $D = qf(t)$, which is obtained for a constant production rate q, can be further analysed into the following

$$\begin{aligned} D = qf(t) &= q[at + qF(t)] \\ &= q[at + b - G(t)] \quad . \quad . \quad . \quad (4) \end{aligned}$$

where a and b are constants and $F(t)$ tends to b and $G(t)$ tends to zero as t tends to infinity. To obtain the physical significance of this substitution, put the values (4) in (1) with $T_1 = 0$; $T_2 = T$ to obtain

$$D = q[aT + G(t - T) - G(t)] \quad . \quad (5)$$

when t tends to infinity, D tends to $qaT = Qa$, where Q is the total production. Hence a is the pressure decline per unit reservoir production.

From equation (4), when t becomes large, D tends to $q(at + b)$. Since at is the permanent pressure drop in the reservoir, b is the proper bottom hole pressure differential for unit production rate.

$G(t)$ is thus the transient caused by the start of the production.

Bottom Hole Pressure Differential

When the bottom hole pressure differential is measured the flow is usually short enough to make the permanent pressure decline aqt due to production negligible. Hence from equation (4)

$$D = q(b - G(t)) \quad . \quad . \quad . \quad (6)$$

The bottom hole pressure differential qb is the limit of D when t becomes large enough to make $G(t)$ negligible. However, this time may in certain cases be very long, and in any case it may be difficult to judge when $G(t)$ in fact became negligible, since the entity measured is D not $G(t)$.

However, if in (5) one finds D_t, D_{T+t}, etc., and at the same time neglect a, one obtains for $0 < t < T$

$$\begin{aligned} D_t &= qG(0) - qG(t) \\ D_{T+t} &= qG(t) - qG(t + T) \\ &\quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \\ D_{(n-1)T+t} &= qG((n - 2)T + t) - qG((n - 1)T + t) \end{aligned}$$

Hence

$$\begin{aligned} D_t + D_{t+T} + \ldots + D_{(n-1)T+t} \\ = qG(0) - qG((n - 1)T + t) \end{aligned}$$

In the limit

$$qG(0) = D_t + D_{t+T} + \quad . \quad . \quad . \quad (7)$$

But from (6), $qG(0) = qb$ = bottom hole pressure differential.

Hence the bottom hole pressure differential is obtainable from equation (7). A special case of equation (7) is when $t = T$, when (7) becomes

Bottom hole pressure differential

$$= D_T + D_{2T} + D_{3T} + \quad (7')$$

If the term at is not neglected, one obtains

Bottom hole pressure differential $= qG(0)$

$$= (D_t - \bar{D}) + (D_{t+T} - \bar{D}) + (D_{t+2T} - \bar{D}) + \ldots$$

where $\bar{D}$ is limit of D as t tends to infinity.

In this particular application this equation is only of academic interest.

The experimental interpretation of measuring bottom hole pressure differential using equation (7) or (7′) is the following:

(1) Measure static bottom hole pressure when well is closed in.

(2) Flow for a short duration, making sure that the flow rate is constant.

(3) Measure the bottom hole flowing pressure at time t after start of flow; and hence find pressure drop D_t.

(4) Close in well at time T after start of flow, (where T is more or equal to t), preferably soon after measurement of flowing bottom hole pressure.

(5) Measure closed-in bottom hole pressures at times $t + T$, $t + 2T$, . . . after start of flow, and thus find the pressure drops D_{t+T}, D_{t+2T}, . . . etc. Keep measuring the pressure drop at equal intervals T until the bottom hole pressure returns to its original value before the start of the experiment.

(6) Sum all the Ds to obtain the bottom hole pressure differential for the given rate of flow.

(7) In the academic case where the pressure comes back to a different value, and thus the reservoir undergoes a finite pressure drop $\overline{D}$ during the experiment, subtract $\overline{D}$ from each D measured, and add those to obtain the actual bottom hole pressure differential.

As a graphical illustration of the above, the pressure build-down for a fractured porous medium has been calculated and plotted in Fig 1. This is the curve $OABCDEF$, where the total bottom hole pressure differential is assumed to be unity and the time scale is arbitrary. The pressure build-up curves have been calculated for different duration of flow T. In fact, curves OAa, OBb, OCc, ODd, OEe correspond to $T = 0{\cdot}2, 0{\cdot}4, 0{\cdot}6, 0{\cdot}8, 1{\cdot}0$ respectively. The following table shows D_n (the pressure drop at time nT) for different values of T and also the sum of Ds for each T.

T	D_1	D_2	D_3	D_4	D_5	D_6	Sum Ds
0·2	0·77229	0·16700	0·04371	0·01250	0·00334	0·00116	1·00000
0·4	0·93929	0·05621	0·0045	—	—	—	1·00000
0·6	0·983	0·017	—	—	—	—	1·00000
0·8	0·9955	0·0045	—	—	—	—	1·00000
1·0	0·99884	0·00116	—	—	—	—	1·00000

The last column illustrates that the sum is independent of the duration of flow and equal to the bottom hole pressure differential.

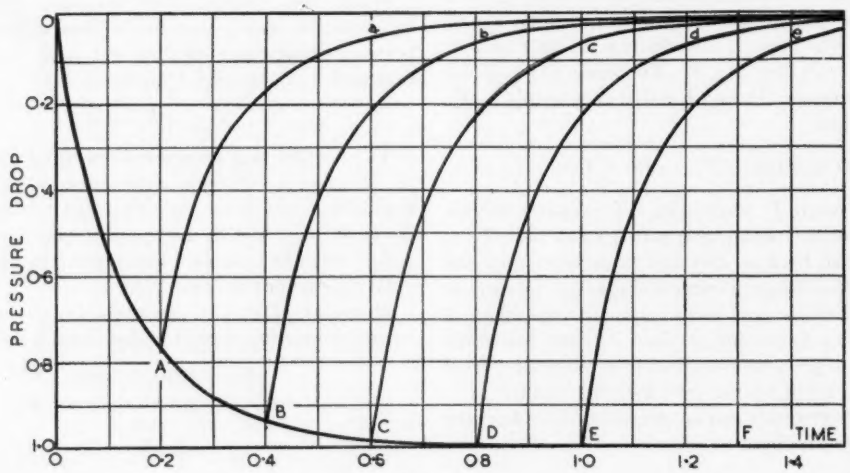

FIG 1

BOTTOM HOLE PRESSURE DIFFERENTIAL

Fractured reservoir 20% of total fluid in fractures.

Analysis of Pressure Decline Curves: Special Case

Divide the past production history into equal intervals of time T such that in each interval the production rate is constant.

Let the production rates be

q_1 from 0 to T

q_2 from T to $2T$

. . . . . .

and q_n from $(n-1)\,T$ to nT.

Then the pressure decline, (see equation (2)) is

$$D = q_1[f(t) - f(t-T)] + q_2[f(t-T) - f(t-2T)] + \ldots + q_n[f(t-(n-1)T) - f(t-nT)]$$

Remembering that $f(t) = 0$ for t less than or equal to 0, one obtains

$$D_1 = q_1 f(T)$$
$$D_2 = q_1[f(2T) - f(T)] + q_2 f(T)$$
$$D_3 = q_1[f(3T) - f(2T)] + q_2[f(2T) - f(T)] + q_3 f(T)$$
$$\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots$$
$$D_n = q_1[f(nT) - f(\overline{n-1}T)] + q_2[f(\overline{n-1}T) - f(\overline{n-2}T)] + \ldots + q_n f(T)$$

when D_r is the pressure decline at time rT.

Using the abbreviation

$$f_r = f(rT) - f((r-1)T) \quad . \quad . \quad (8)$$

the above equations reduce to

$$D_1 = q_1 f_1$$
$$D_2 = q_2 f_1 + q_1 f_2$$
$$D_3 = q_3 f_1 + q_2 f_2 + q_1 f_3$$
$$D_4 = q_4 f_1 + q_3 f_2 + q_2 f_3 + q_1 f_4$$
$$\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots$$
$$D_n = q_n f_1 + q_{n-1} f_2 + \ldots + \ldots + q_1 f_n \; . \quad (9)$$

If the Ds and qs are known accurately, then f_1 is obtained by solving the first equation, this value is substituted in the second equation, and the value of f_2 is found, etc., until finally f_n is found. Once all the f_rs are found, and since $f(0) = 0$, equation (8) gives the values of $f(rT)$ for $r = 1, 2, \ldots n$; *i.e.* the pure pressure decline curve is obtained. When the reservoir has been produced long enough, the transient in $f(t)$ (see equation (2)) would have vanished and f_n becomes a constant with respect to n. Thus f_n becomes known for all n, and equation (9) can be used to predict the pressure drop for any production history directly, provided its production rates can be treated as constant during the unit intervals T. Otherwise, f_n can be plotted against n, and the values of f_n be found graphically or otherwise and these inserted in (2) to find the appropriate pressure drops.

A model of the reservoir is thus unnecessary for purposes of prediction. However, when such a model becomes necessary, the pure curve $f(t)$ can be used to determine the constants in the model of the reservoir.

The difficulty in applying equation (9) lies in the inaccuracies inherent in observation of the pressure drop D and rate of production q. A method of trial and error, which may need the services of a digital computer, may be necessary to find a series of regularly varying f_r that tend towards a limit which best fit equation (9).

Another method of approach is to substitute for f_r the value (*cf* equation (4))

$$f_r = aT + F_n$$
where $$F_n = F(nT) - F((n-1)T)$$

Thus equations (9) become

$$D_1 = aQ_1 + q_1 F_1$$
$$D_2 = aQ_2 + q_2 F_1 + q_1 F_2$$
$$D_3 = aQ_3 + q_3 F_1 + q_2 F_2 + q_1 F_3$$
$$\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots$$
$$D_n = aQ_n + q_n F_1 + q_{n-1} F_2 + \ldots + q_1 F_n \quad (10)$$

where Q_r is the total production up to time rT.

The part aQ represents the permanent pressure decline in the reservoir due to the production Q, and the rest is the transient due to variation of production rates.

Analysis of Pressure Decline: General Case

When the restriction that the production rate is the stepwise function referred to in the previous paragraph is removed the pressure decline p is

$$p = \int_o^t q(t-u) f'(u) du \quad . \quad . \quad (11)$$

deducible from equation (3) by the substitution $v = t - u$; and the pure decline curve is the solution of this integral equation. Integrating (11) by parts one obtains

$$p = q(o) f(t) + \int_o^t q'(t-u) f(u) du \; . \quad (12)$$

Hence

$$f(t) = \frac{q}{q(o)} - \frac{1}{q(o)} \int_o^t q'(t-u) f(u) du$$

or

$$f(t) = g_o(t) + \int_o^t k(t,u)\, f(u)\, du \quad . \quad . \quad (13)$$

where $g_o(t)$ and $k(t,u)$ are $\frac{p}{q(o)}$ and $\frac{-q'(t-u)}{q(o)}$ respectively. The solution of equation (13), which is equivalent to (11), is possible. As a matter of fact the infinite series

$$f(t) = g_o(t) + g_1(t) + g_2(t) \ldots \; . \quad (14)$$

where

$$g_n(t) = \int_o^t k(t,u) g_{n-1}(u) du \quad . \quad . \quad . \quad (15)$$

is convergent and satisfies the integral equation (13), and thus is the required solution. To prove the convergence of (14) it is noticed that both k and g_o are bounded, *i.e.* there exist numbers M, N such that

$$|k| < M \text{ and } |g_o| < N$$

hence from (15)

$$|g_n| < M \int_o^t |g_{n-1}| du$$

and it can be proved by induction from the above equation that

$$|g_n| < NM^n \frac{t^n}{n!}$$

and thus $$|f| \leqslant \Sigma |g_n| < \Sigma NM^n \frac{t^n}{n!}$$

but the right hand side is convergent (exponential series), hence the series for f_n is absolutely convergent.

Equation (15) can be transformed by integrating by parts of the recurrence relation

$$g_n(t) = -\int_o^t \frac{q'(t-u)}{q(o)} g_{n-1}(u)du = +\frac{1}{q(o)}\int_o^t \frac{\partial}{\partial u} q(t-u)$$

$$g_{n-1}(u)du = g_{n-1}(t) - \int_o^t \frac{q(t-u)}{q(o)} g'_{n-1}(u)du \quad (16)$$

which is a more convenient form for computation.

When the production rate is constant, $k = o$, and hence $g_n = o$ for n more than zero, and thus

$$f(t) = g_o = \frac{p}{q(o)}$$

which is what one expects.

The drawback to this analysis is that there is an over-emphasis on the initial production rate $q(o)$, and if that happens to be in error, this error is carried through the calculation, since it occurs in the denominator in both equations (15) and (16). On the other hand, one can look on $q(o)$ as a parameter, and obtain solution (14) for different values of this parameter and choose that solution which gives the best fit when substituted back in equation (11). A thorough investigation of this point deserves notice and shall be attempted in the near future. An important point in favour of this solution is that one knows that it is convergent.

More general solutions of the integral equation (11) can be obtained which are free from the above drawback of the over-emphasis on the initial production rate, but which are conditional; *i.e.* they are solutions only in so far that they are convergent. Consider any weighing function $Z(t)$, and the series of function $G_n(t)$, where

$$G_n(t) = G_{n-1}(t) - Z(t)\int_o^t q(t-u)G'_{n-1}(u)du \quad (17)$$

and $G_o(t) = Z(t)$p.

Then if the series

$$f(t) = G_o + G_1 + G_2 + \ldots \quad . \quad (18)$$

is convergent, and thus the function $f(t)$ exists, then this function is a solution of the integral equation (11). To prove that, add the equation (17) for $n = 1, 2, \ldots$, to obtain

$$G_1 + G_2 + \ldots = G_o + G_1 + \ldots - Z(t)\int_o^t q(t-u)/(G'_o + G'_1 + G'_2 + \ldots)du$$

or

$$f - G_o = f - Z(t)\int_o^t q(t-u)f'(u)du$$

But $$G_o = Zp$$

hence $$G_o = Zp = Z\int_o^t q(t-u)f'(u)du$$

which is equation (11) multiplied by Z.

So far, Z is any function of t which ensures the convergence of (19), but from general considerations its choice should be such as to make the convergence as fast as possible, and thus when the production rate is constant q, G_o should reduce to p/q, or the value of $Z(t)$ for constant production should be $\frac{1}{q}$.

Among the possible functions Z that satisfy this general condition the two simplest are

$$Z = \frac{1}{q(o)} \text{ and } Z = \frac{t}{Q(t)}$$

The first choice of Z gives us the solution (14) which was discussed above. The second choice makes (17) read

$$G_n(t) = G_{n-1}(t) - \frac{t}{Q(t)}\int_o^t q(t-u)G'_{n-1}(u)du. \quad (20)$$

In effect here $\frac{1}{Z}$ instead of being the initial production rate becomes the average production rate since the start of production.

CONCLUSION AND SUGGESTIONS

The analysis of pressure decline in general leads to two applications: (1) the estimation of bottom hole differential pressures; and (2) the construction of a pressure decline curve for constant production rate from an actually observed curve for varying production rates, and thus to predicting pressure decline for any given production. The analysis, though theoretical in nature, was general and depended on very few plausible assumptions, and thus should have wide applicability.

(1) As far as the bottom hole pressure differential is concerned it is suggested that experiments should be carried out to test the truth of the theory and to establish a routine for such measurements. A series of experiments should be carried out in which the bottom hole pressure differential, D, of a well is measured for a constant rate of flow, q, using the

procedure outlined above where the well is allowed to flow for time T. The experiment is repeated for the same q and different T. The theory shows that the bottom hole pressure differential should not depend on T, as long as q is the same. Practically, there may be some dependence for small T because of the effects of after production, and of the difficulty of keeping q constant. The result of these experiments would show what is the least practical value of T which can be used to give consistent results.

(2) The analysis of pressure decline curves by this method presents the arithmetical problem of solving equation (9) or (15) and (16). It is suggested that a thorough investigation be carried out, possibly with the help of a digital computer, into these computational problems, especially on the effect of small variations of the measured experimental data on the final curve.

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DENSITY OF RESERVOIR CRUDE OIL, WITH REFERENCE TO THE CALCULATION OF BOTTOM HOLE PRESSURES *

By D. E. BUNYAN,† T. I. SHAFIQ,† and G. J. M. MANASTERSKI †

SUMMARY

By measurement of the density of reservoir oil under the conditions found in a closed-in well, bottom hole pressures may be calculated accurately from measurements of surface pressures (minimum closed-in pressures). The determinations of density are carried out with the aid of conventional p–v–t equipment, with the addition of an extra pressure-cell for weighing. Weighings are done on a bullion balance. The accuracy of the method is discussed, and it is shown that the probable error in the pressure computed at the bottom of a 3000-ft well is $\pm 0{\cdot}25$ p.s.i. or less.

To ascertain the pressure in an oil reservoir, it may often be simpler to measure, in a static well, either the pressure at the surface or, in a very low pressure field, the level of the free oil, than it is to employ any of the various devices for measuring sub-surface pressures. The surface measurement is not of value, however, unless it can be used for deriving a reasonably accurate sub-surface pressure, and for this purpose the density of the oil in the well must be known, under the conditions prevailing there. W. J. Baker ‡ has given a solution to the problem, and the work to be described here is largely an adaptation of his work. The method of measuring the density itself will be discussed, and also the method of employing the results in obtaining bottom hole pressures. First, however, the method used in determining the surface pressure must be considered briefly, in order to evaluate what range of conditions the density measurements must embrace. For this, the practices at the Kirkuk field will serve as illustration.

The surface pressure is measured on a closed-in well, after the column of oil in the well has been conditioned by bleeding off gas as it is formed. A strict routine is observed in this procedure, to ensure that the conditioning will as nearly as possible be the same on successive occasions. The surface pressure (or the level of the free oil in the open well) is then measured at a fixed time (24 hr $\pm$ 1 hr) after the initial closing-in. The result is stated as a gauge pressure in pounds per square inch, at a common reference level for all wells; in the conversion from the level of measurement to the level of reference, a pressure-gradient in oil of 0·348 p.s.i/ft is assumed, based on earlier but somewhat dubious measurements. The pressure so computed is called the minimum closed-in pressure (M.C.I.P.). More reliance is placed on measurements made at a producing well, shut down only for the conditioning and the measurement, than on those at an observation well, where prolonged shut-down may age the oil in ways difficult to predict.

The pressure, temperature, and degree of saturation of the oil in the column therefore range from one set of limiting values at the surface to another set deep in the reservoir, and the density varies accordingly. Kirkuk values will serve as examples. In temperature, the range is from a mean value of about 80° F, just below the surface of the earth, to something between 100° and 120° F in the reservoir. The pressure will range between zero, at the top of a closed-in, bled-off well, to around 1500 p.s.i.g. The saturation pressure in the oil near the top of a conditioned well will be the same as the ambient pressure, and in the

* MS received 17 June 1957.
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‡ Baker, W. J. *Proc. Third World Petrol. Congr.*, 1951, **2**, 474–81.

reservoir itself will be between 550 p.s.i.g. and just under 400 p.s.i.g. Hence, to be relevant to a study of the Kirkuk reservoir, the measurements of density must cover a range of conditions from atmospheric pressure (at zero saturation pressure and temperature of 80° F) to pressures of 1500 p.s.i.g. (at saturation pressures between 350 and 550 p.s.i.g. and temperatures between 100° and 120° F).

Saturation pressure can be varied by collecting unsaturated oil from a point deep in the reservoir and bleeding off gas, in the laboratory, until the degree of degassing required is achieved. Alternatively, oil can be collected from a static, bled-off well, from points at various heights above the saturation level so selected as to yield oils of the required range of saturation pressures. The second course was the one followed by Baker, and also in the current set of experiments, while the first course was followed in earlier measurements made in Kirkuk. The differences between the two sets of results will be indicated later.

In his measurements Baker used the sample-taker in which the oil had been collected as his "*p–v–t* cell." The earlier measurements of density made in Kirkuk followed the same method, but the technique is cumbersome. The pressure can be independently varied only by injecting water into the sample-taker, and the volume changes determined only by weighing. The frequent making and breaking of connexions makes for many leaks, and weighing is a slow and tedious method of finding volume. Standard *p–v–t* equipment and methods were therefore used in later work, with certain changes made necessary by the special requirements of the experiment.

Samples were collected in a flush-through type of sample-taker (the kind made by the Coleman Instrument Manufacturing Company) with a capacity of 750 cc. For the main part of the experiment, a number of samples were collected from various depths in a single well on the same day, within a few hours either way of the 24th hour from the initial closing-in—the well having been given the standard conditioning as for M.C.I.P. measurements. The well chosen was a producer, the one on which the best continuous series of measurements had been made in the past, and to which measurements on other wells are correlated. The depths of sampling were selected so as to give a good range of saturation pressures. Additional samples had to be taken from other wells, to obtain oil of higher saturation pressure than that now obtained from the "reference" well (but which was needed for evaluating earlier readings on the well, made when the saturation pressure was higher). No sample could be taken from near the surface of the oil, as the pressure there is zero and the valves in the sample-taker will not close satisfactorily, nor remain closed, without some excess of pressure on the inside. Instead, a sample was collected from a little below the surface, where the oil was gassy enough to maintain a pressure in the sample-taker, under the temperature of storage, of around 25 p.s.i.g. This sample was subsequently degassed in the laboratory, at 90° F, to atmospheric pressure.

The actual measurements were carried out in standard *p–v–t* equipment—pressure cell, constant-temperature bath, and mercury pump—manufactured by Engineering Products Inc. (with pressure-gauges by Heise), plus an additional pressure-cell made in our own workshops. The samples as collected were stored in the original sample-takers until required. On each, the density was measured at 1000 p.s.i.g. and 90° F, the saturation pressure was measured at three temperatures (one of them being 90° F), and the volumetric behaviour at the three temperatures and over a range of pressures was investigated. With the density known at the reference conditions of 1000 p.s.i.g/90° F, and the volume known at these and a range of other conditions, the density at the other conditions is readily computed. The procedure in dealing with the successive samples changed somewhat as the technique was improved, but the latest is more or less as described below.

From the oil in the sample-taker, some 50–100 cc are transferred to the *p–v* cell. The saturation pressure at one of the three temperatures is measured, and this fraction then thrown away.

The extra pressure-cell, which for brevity will be denoted as the "density cell," is thoroughly cleaned, dried, evacuated, and weighed on the bullion balance. About 250 cc of oil are transferred to the *p–v* cell, and brought to a temperature of 90° F and a pressure of 1000 p.s.i.g. The density cell is then connected with a short length of tubing to the *p–v* cell, and density cell and tubing both evacuated. With the valve on the density cell closed, the tubing is filled with oil from the *p–v* cell, which is still kept at the standard conditions, and the reading of the mercury pump is noted. About 200 cc of oil are now pumped from *p–v* cell to density cell, and the reading of the mercury pump again noted. From the difference between initial and final readings can be found the volume at 1000 p.s.i.g. and 90° F of the oil which went into the density cell (and is, of course, no longer under those conditions), of which the mass will not have been affected by the operation. The density cell is then cleaned and dried externally, and again weighed; the difference between this and the initial weight gives the mass of oil in the cell. From this mass, and the volume occupied by this oil at the reference conditions, the density of the oil at those conditions is computed.

The oil in the density cell is discarded. Of the oil remaining in the *p–v* cell, the saturation pressure is measured at one temperature, and the oil is discarded.

A further 250 cc of oil are then transferred to the *p–v* cell, and the volume at 1000 p.s.i.g. and 90° F is found. The volume is then measured under all the

conditions of temperature and pressure that are of interest in the particular case—the three temperatures at which the saturation pressure has or will be measured, and four or five pressures at each temperature. The sample is then returned to 1000 p.s.i.g/90° F (which provides the opportunity of measuring the volume under reference conditions if this was not done initially), and the transfer of 200 cc from p–v cell to density cell is made, for a check on the value of density that was first found.

The oil in the density cell is discarded. The saturation pressure of the oil (50 cc) remaining in the p–v cell is measured at the third of the three temperatures of interest, and this oil, too, is discarded.

If any oil remains in the sample-taker, it is transferred to the p–v cell and the saturation pressure is checked, at one of the temperatures previously measured.

In all the operations, care is taken not to let the pressure of the oil drop below the saturation pressure, except in the actual measurements of this property. This is because, with this equipment, oil and gas cannot be recombined, after having been allowed to separate, without large errors in volume being observed. The errors are believed to be caused by the trapping of gas in the oil–mercury emulsion which is formed when the p–v cell containing oil and mercury is shaken.

The observations are repeated on each sample in turn, and so the density of the oil under a wide range of temperatures and pressures is found, for oils of varying gas content. The results of this are given in Table I. Before discussing the results, some consideration should be given to the sources of error in the measurements. The smallness of the changes in density produced by small changes in temperature, pressure, and saturation pressure, together with the closeness with which these functions can be controlled or measured ($\pm 0{\cdot}1°$ F, ± 2 p.s.i.), makes these relatively small sources of error. The volume can be measured with an error of around $\pm 0{\cdot}005$ cc, which again contributes but little to the total error. The largest source of error is therefore the weighing.

The weight of the oil is found as a difference between the filled weight and the empty weight of the density cell. The cell being necessarily somewhat heavy—in this case it weighed just over 6 kg—only a large, rather insensitive balance can be used, such as the Oertling bullion balance which was employed in this work. As the oil will weigh something like 200 g or less, great care is required in the use of the balance, in order not to sacrifice accuracy too seriously. The performance of the balance was therefore checked by weighing the weights against each other in a cumulative series of measurements which found out the sensitivity of the balance, assessed the change in balance-point with increase in load, determined the repeatability of the measurements as given statistically by the standard deviation of a series of readings, and also calibrated the weights in terms of one weight chosen as standard.

A shift in balance-point with increase in load was found, and compensated by double-weighings, with the cell and the weights switched to opposite pans between the two weighings of each pair.

Calibrating the weights did not sufficiently reduce the uncertainty in the values of the larger weights. Therefore care was taken to employ the same large weights in all weighings. The weight of the oil, found as a difference, was then a function only of the values of the smaller, more accurately known weights.

The sensitivity of the balance was found to be 0·12 g per division of the pointer scale. As the equilibrium position of the pointer can be estimated by the free-swing method to within one-tenth of a division, the error in a single balancing is therefore $\pm 0{\cdot}01$ g. Statistical evaluation of the errors in a series of weighings gave a standard deviation about one-third of the above. As the final weight of the oil is the difference between two double weighings, the error in this weight is of the order of $\pm 0{\cdot}015$ g or $\pm 0{\cdot}02$ g. The contribution of such accidental factors as dirt on the cell, or in the fittings on the cell, does not seem to have increased the error much above this value.

Taking the weighing error as $\pm 0{\cdot}02$ g, and taking the weight of the oil as, say, 160 g, then for an oil density of around 0·8 g/cc the error in the density will be $\pm 0{\cdot}0001$ g/cc. Allowing the other sources of error to increase this slightly, consider the error in density as 0·00012 g/cc. The authors are interested, eventually, in computing a bottom hole pressure; if the error occurred in the same sense through the whole series of measurements (an unlikely event), then the calculations of a bottom hole pressure from an assumed accurate surface pressure would, for a column of 3000 ft, be in error by $\pm 0{\cdot}16$ p.s.i. Of course, in practice, uncertainties as to the temperature and saturation pressure of the oil in the well will increase the error, but even so, the method would appear to permit the calculation of the pressure at the bottom of a 3000-ft well with a probable error of not more than $\pm 0{\cdot}25$ p.s.i.

Refer again to Table I, where the density is tabulated as a function of saturation pressure, temperature, and pressure. For use in calculations of pressure, the data must be smoothed, which can be done either graphically or algebraically; it was found convenient to fit an empirical algebraic equation to the data. A reasonably good fit was found for the following equation:

$$\rho = 0{\cdot}82816 + 0{\cdot}03697e^{-0{\cdot}002552s} - (4{\cdot}106 + 0{\cdot}000471s) \times 10^{-4}t + (3{\cdot}68 + 0{\cdot}00113s + 0{\cdot}0147t) \times 10^{-6}p + \frac{4343}{1{\cdot}467 \times 10^{6} + (s - 20)^{4}} \quad (1)$$

where ρ is the density of the oil in g/cc;
s is the saturation pressure (p.s.i.g.) as measured at 90° F;
p is the applied pressure, in p.s.i.g.;
t is the temperature, in ° F;

(The last term in Equation (1) can be ignored at saturation pressures greater than 100 p.s.i.g.)

The last column of Table I shows the deviations of the experimental values from the values computed by Equation (1). It will be seen that the errors tend to be of the same sense and magnitude within the set of readings for any given saturation pressure, *i.e.* for any particular sample. This is what would be expected if either the weighing or the saturation-pressure determination were the largest source of error; the trend of previous argument was that the weighing was probably at fault. The standard deviation computed for the whole assemblage is ±0·000107 g/cc. If the two values which display errors four and five times this are rejected, according to normal practice, the standard deviation then works out at ±0·000074 g/cc. Either value agrees well enough with the error anticipated on the basis of the accuracy in weighing.

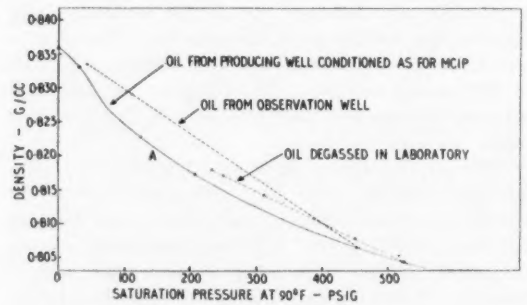

FIG 1

DENSITY OF KIRKUK CRUDE OIL

Density at 1000 p.s.i.g. and 90°F as a function of saturation pressure

Graph A = Equation (1)

In Fig 1 the density at 1000 p.s.i.g. applied pressure and 90° F is graphed as a function of saturation pressure, the experimental values being shown as circled dots, and the graph of Equation (1) being drawn as a solid line. Also shown are values from an earlier, incomplete set of measurements on oils taken from various depths in an observation well, from which no flow of oil had occurred for several years. The third set of values shown are for a sample which was taken from the region of the reservoir where saturation pressure is high, and which was then degassed in the laboratory, the density being measured at each stage. In these latter two sets the density at the highest saturation pressure falls on the line of Equation (1), showing that the measurements prior

TABLE I

Saturation pressure at ambient temperature, p.s.i.g.	Temperature, ° F	Density at 1000/90 g/cc	Pressure, p.s.i.g.	Volume, cc	Density, g/cc	Errors, g/cc × 10⁴ +	Errors, g/cc × 10⁴ −
0	80	(1)	526	201·833	0·83732	19	—
		0·8359	356	201·982	0·83670	—	2
		(2)	200	202·171	0·83592	—	—
		0·8359	52	202·470	0·83468	52	—
0	90	(3)	1000	202·192	0·83583	1	—
		0·8357	533	202·746	0·83355	—	4
			359	202·957	0·83267	—	3
			198	203·160	0·83185	—	1
			50	203·360	0·83103	7	—
	100		1000	203·139	0·83193	—	4
			526	203·713	0·82959	—	14
			357	203·932	0·82870	—	12
			201	204·135	0·82787	—	9
			54	204·374	0·82691	11	—
28	80	(1)	1000	272·018	0·83714	—	7
		0·8330	464	272·859	0·83456	—	11
			352	273·038	0·83402	—	12
		(2)	254	273·199	0·83352	—	10
		0·8331	153	273·234	0·83342	—	49
32	90		1000	273·354	0·83305	5	—
			464	274·223	0·83041	—	1
			352	274·406	0·82986	—	3
			251	274·578	0·82934	—	2
			148	274·751	0·82882	—	1
35	100		1000	274·640	0·82915	—	2
			463	275·542	0·82644	—	10
			355	275·729	0·82588	—	10
			252	275·912	0·82533	—	8
			152	276·097	0·82478	—	5
120	80	(1)	1000	274·980	0·82677	—	2
		0·8227	499	275·813	0·82427	—	2
			402	275·983	0·82376	—	—
		(2)	302	276·159	0·82324	2	—
		0·8228	202	276·265	0·82292	—	16
125	90	(3)	1000	276·317	0·82277	—	4
		0·8228	550	277·125	0·82037	—	6
			410	277·348	0·81971	—	2
			299	277·551	0·81911	1	—
			200	277·733	0·81857	4	—
130	100		1000	277·665	0·81877	—	6
			528	278·529	0·81623	—	2
			415	278·744	0·81560	2	—
			303	278·955	0·81499	3	—
			204	279·138	0·81445	5	—
197	80		999	273·583	0·82136	6	—
			605	274·251	0·81936	5	—
			501	274·438	0·81880	8	—
			399	274·616	0·81827	9	—
			302	274·795	0·81773	14	—
207	90	(1)	999	274·943	0·81729	7	—
		0·8172	605	275·643	0·81522	8	—
			411	276·000	0·81416	12	—
		(2)	297	276·214	0·81353	15	—
		0·8174	256	276·293	0·81330	17	—
218	100		999	276·315	0·81323	7	—
			602	277·050	0·81108	8	—
			500	277·239	0·81052	10	—
			404	277·422	0·80999	11	—
			306	277·615	0·80943	14	—
452	90	(1)	996	257·396	0·80648	—	4
		0·80662	850	257·652	0·80568	—	5
		(2)	698	257·921	0·80484	—	4
		0·80635	595	258·106	0·80426	—	3
478	105		996	259·402	0·80024	—	5
			844	259·686	0·79937	—	6
			700	259·954	0·79854	—	6
			592	260·163	0·79790	—	4
505	120		996	261·445	0·79399	—	6
			854	261·726	0·79313	—	5
			700	262·029	0·79222	—	5
			601	262·231	0·79161	—	3
524	90	(1)	1147	268·786	0·80504	8	—
		0·8042	1007	269·039	0·80428	5	—
		(2)	867	269·310	0·80347	8	—
		0·8043	702	269·626	0·80253	10	—
553	105		1147	270·886	0·79880	4	—
			1006	271·163	0·79798	4	—
			851	271·464	0·79710	2	—
			702	271·757	0·79624	1	—
583	120		1148	273·026	0·79254	3	—
			1005	273·321	0·79166	5	—
			847	273·646	0·79074	1	—
			710	273·930	0·78992	—	—

to degassing are consistent with the present set. The density at any particular saturation pressure is therefore very dependent on the method of degassing, as is shown by the way the sets of values diverge at the lower pressures.

Having obtained an expression for the density of oil under a range of conditions which includes those in a well conditioned for measurement of minimum closed-in pressure, we need only a little further information in order to compute the bottom-hole pressure from the M.C.I.P. The temperature in the well must be measured, also with the well conditioned as for an observation of M.C.I.P., at a sufficient number

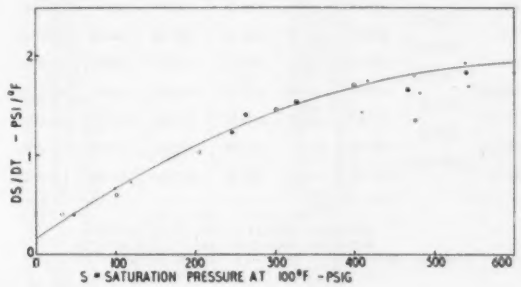

FIG 2

RATE OF CHANGE OF SATURATION PRESSURE WITH TEMPERATURE AS A FUNCTION OF SATURATION PRESSURE AT 100°F

$DS/DT = 1{\cdot}95–4{\cdot}24\ (650\text{-}S)^2 \times 10^{-6}$

of depths that a reasonably accurate temperature–depth curve can be drawn. The saturation-pressure of the oil produced by the well must be known, and also, of course, the M.C.I.P.

The variation of saturation-pressure with temperature must be studied. The evidence of the present study as well as of earlier work is that, to a sufficient degree of accuracy, the saturation pressure as measured varies linearly with the temperature of measurement, but that the slope of this variation is a function of saturation pressure. This function takes the form shown in Fig 2, where results of a number of investigations are assembled. The line drawn fits this equation:

$$\frac{ds}{dt} = 1{\cdot}95 - 4{\cdot}24\ (650 - s)^2 \times 10^{-6} \qquad (2)$$

where $\frac{ds}{dt}$ is the derivative of saturation pressure with respect to temperature (p.s.i/°F);

s is the saturation pressure (p.s.i.g.) measured at 100°F;

t is the temperature in °F.

The line is not fitted by statistical methods to the whole set of points, but is drawn through what seemed on mainly subjective grounds to be the most reliable ones. The scatter being so great, the method is as reliable as the data.

We are now in a position to calculate the bottom hole pressure. We take the M.C.I.P. and remove the correction to common reference level, to obtain the pressure at the top of the column of oil in the particular well. From the temperature measurements, presumably smoothed algebraically, we have the temperature at this point. The saturation pressure at ambient

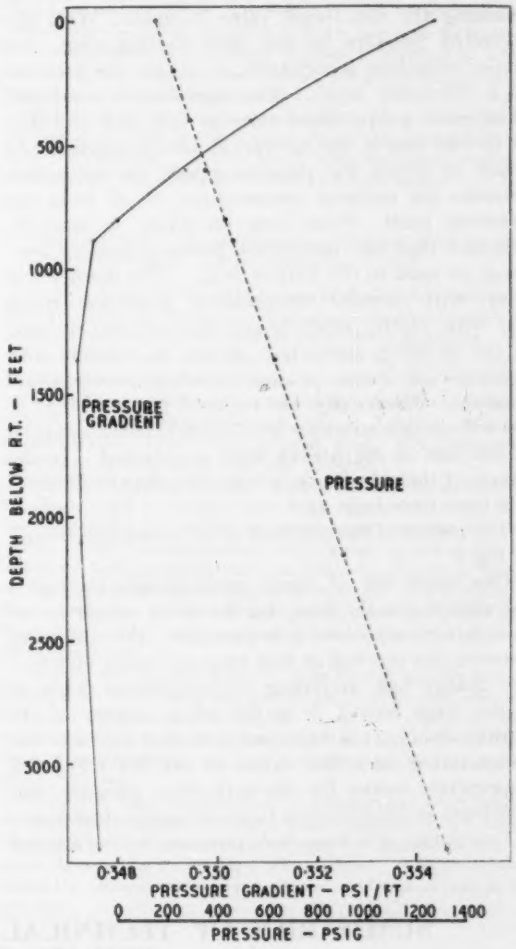

FIG 3

GRAPH OF PRESSURE AND PRESSURE GRADIENT AGAINST DEPTH

M.C.I.P. = 155 p.s.i.g.
Saturation pressure = 476 p.s.i.g. at 100°F
457·8 p.s.i.g. at 90°F

temperature is equal to the pressure itself in this, the saturated region of the column, and the saturation pressure at reference temperature (90° F) is obtained from it by use of either Fig 2 or Equation (2). From the pressure, temperature, and saturation pressure,

the density of the oil is computed, using Equation (1), and, from this, the pressure gradient at the top of the oil column. With the gradient so found, we compute the pressure at a convenient interval of depth (say, 200 ft) farther down, and by a similar set of calculations find the gradient here. The mean of the two gradients is then used to find a corrected value for the pressure at the lower depth; if a short enough interval is selected it will not be necessary to recompute the gradient at the lower point, as the error involved in retaining the first-found value is small. With the corrected pressure as the new starting-point, the whole procedure is repeated, to obtain the pressure at a still lower depth. The repetition is continued until, with a final depth-interval that will probably be of odd length, the saturation level is reached, the depth at which the pressure equals the saturation pressure (at ambient temperature) of oil from the reservoir itself. From here on down, it must be assumed that the saturation pressure remains constant (at least in the Kirkuk case). The density will vary—with normal temperature gradients—much less with depth; much larger intervals can be used in the iterative calculation, chosen to coincide with intervals of more or less constant temperature gradient. Eventually, the required pressure, that at the sub-surface reference level (2200 ft below sea-level in the case of the Kirkuk field) is obtained. In the course of the calculation, a table like that in Table II will have been built up.

The results of the calculation are shown graphically in Fig 3.

One would not, of course, wish to carry through a calculation like the foregoing for every conversion of a surface to a bottom hole pressure. The difference between the two will in fact vary extremely slowly—the change has, according to calculations made as above, been only 4 lb in the whole history of the Kirkuk field. It is therefore sufficient to make the computation for a few values of the M.C.I.P., with appropriate values for the saturation pressure, and construct an interpolation table for rapid calculation.

Calculation of bottom hole pressures by the method just described gives results consistent with measurements by bottom hole gauges. As the calculation method should be somewhat more accurate than the other, a fairly careful and cunning set of tests would have to be made if direct measurements were to provide a check on the calculations. Since completion of the density measurements, opportunities of making tests on producing wells have been somewhat limited. It is hoped, however, that the results can be corroborated by bottom hole measurements in due time. Meanwhile it can be stated that the method gives every evidence of being a good one for obtaining values for bottom hole pressures from surface measurements.

TABLE II

Depth below R.T., ft	Depth interval, ft	Pressure, p.s.i.g.	Temperature, ° F	Saturation pressure, p.s.i.g.	Corrected pressure, p.s.i.g.	Corrected saturation pressure, p.s.i.g.	Pressure gradient, p.s.i/ft
0		155·00	86·72	157·98			0·35439
	200·00						
200		225·88	88·34	227·94	225·68	227·74	0·35239
	200·00						
400		296·16	89·96	296·21	295·99	296·04	0·35069
	200·00						
600		366·13	91·58	363·53	365·99	363·39	0·34929
	200·00						
800		435·85	93·21	430·13	435·72	430·00	0·34802
	83·84						
883·84		464·88	93·89	457·80	464·88	457·80	0·34755
	483·66						
1367·5		632·98	97·81	457·80	632·90	457·80	0·34724
	785·00						
2152·5		905·48	101·15	457·80	905·50	457·80	0·34729
	1196·00						
3348·5		1320·86	105·04	457·80	1321·06	457·80	0·34763

M.C.I.P. = 155·0 p.s.i.g.
Saturation pressure = 476 p.s.i.g. at 100° F
Saturation pressure = 457·8 p.s.i.g. at 90° F
Pressure at 2200 ft below sea-level = 1321·06 p.s.i.g.
B.H.P. − M.C.I.P. = 1166·06 p.s.i.g.

ACKNOWLEDGMENTS

Thanks are due to the Iraq Petroleum Company Ltd for permission to present this paper, and to H. A. Freeman for instigating the work described and giving valuable advice and encouragement in the course of it.

SUMMARIES OF TECHNICAL NOTES PRESENTED AT THE KUWAIT SYMPOSIUM

SOME OPERATING DIFFICULTIES EXPERIENCED WITH THE RUSKA SUBSURFACE SAMPLER. P. D. O'Connell. (Qatar Petroleum Co. Ltd.)

The Ruska subsurface sampler is of the flush type. The valves of the sample container are " O-ring " sealed, and are closed by the pressure of the well fluid after the timing clock has released the tripping mechanism (Fig 1). The sample is transferred to the shipping bottle by a closed circuit after mercury has been pumped into the shipping bottle to raise the pressure to that of the sample-taker, thereby opening the valves and allowing displacement of the sample by gravity.

Difficulties have arisen in the operation of the sampler. The commonest trouble has been the obtaining of samples with a low opening pressure when connected to the shipping bottle, pointing to leakage past a valve. This was overcome by machining the " O-ring " groove deeper, and by making the diameter of the part of the valve above the ring slightly less than that of the part below, so that the lower part of the valve seats first.

Pipe scale and other solids sometimes prevented valves from seating properly. A second filter fitted to the upper valve gave satisfactory results. Some non-representative samples were the consequence of premature operation of the

tripping mechanism due to vibration. Careful running of the instrument removed this trouble when the sampler was new, but weakening of the hammer lock spring on continued use made the device more prone to premature tripping. It is hoped that springs of Ni-Span C will reduce this weakening, which is thought to be a temperature effect.

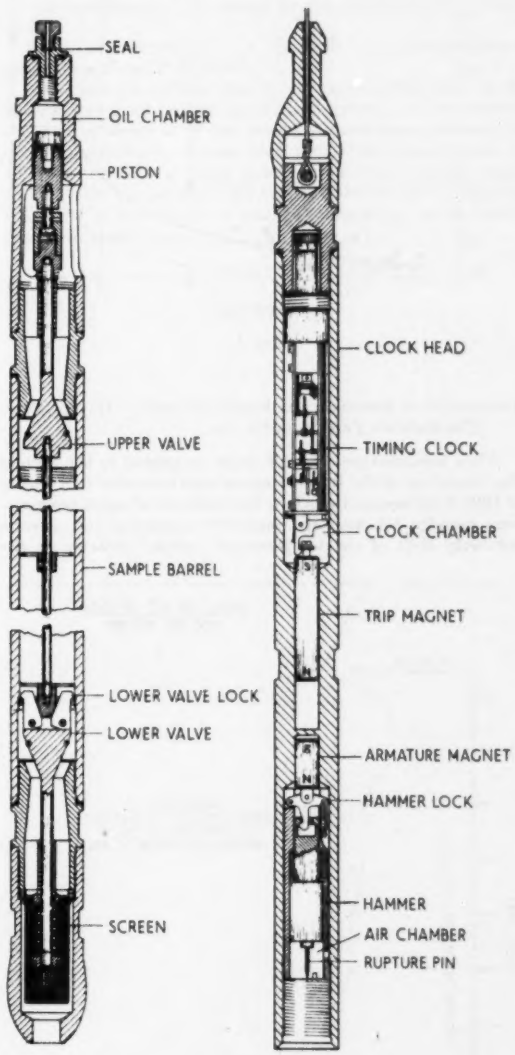

FIG 1

In the transfer operation the lower valve did not open fully, and the upper valve tended to close under the transfer flow. These troubles were overcome by removing the closure spring of the lower valve before fitting the transfer head, and by installing a stronger spring in the upper transfer head.

The various modifications improved the reliability of the instrument, and led to the obtaining of satisfactory subsurface samples in nine cases out of ten.

DRILL-STEM TESTING RESULTS. P. D. O'Connell. (Qatar Petroleum Co. Ltd.)

Conventional Johnston tester equipment has been used in Qatar, generally comprising packer, and equilizing, retaining, and trip valves, with other auxiliary components as required. Single hook-wall packers have usually been employed in casing tests, and dual wall packers in open-hole tests. Water cushions have ranged from zero to a few thousand feet. Open-hole anchors up to 100 ft long have been made up from drill collars. Normal packer setting times have been one or two hours; however, as much as 30 hours has been used in open-hole and 55 hours in casing. Weight on packers has varied from 13,000 to 90,000 lb. There has been no trouble from stuck packers. Pressure recorders have rarely been employed, but the Johnston recorder has given more reliable service than the Amerada recorder.

Casing packer tests have almost always been mechanically successful, whereas nearly 50 per cent of the open-hole tests have been failures. Most of the failures have been with the packers, and have been attributed to excess weight.

A NOTE ON THE USE OF BINACAL OIL-BASE MUD IN THE DUKHAN FIELD. Qatar Petroleum Co. Ltd.

The No. 3 and the No. 4 limestones of the Dukhan field have been cored in a few wells with Binacal oil-base mud in order to obtain cores for accurate connate-water determinations. 80-brl batches of mud were prepared on the rig site by mixing 47 brl of gas oil, 8·4 tons of Binacal (a coarse powder consisting of a mixture of blown bitumen, calcium naphthenate compound, naphthenic acids, calcium oxide, and chalk), and 4 brl of fresh water. This gave 75-lb/cu. ft. mud. Weighting with barytes or calcium carbonate was possible.

The mud was very stable. When necessary oil was added to check rises in weight, and Bitca (blown bitumen and calcium oxide) was added to compensate for the diluting effects of oil. Contamination with water caused a rise in viscosity, but experience and restrictions in the use of water on the rig enabled this trouble to be avoided. The initial mix had a viscosity of 85–100 sec (Marsh), and an initial gel strength of 2–4 g and a 10-min gel strength of 5–8 g; the filtrate was zero.

Drilling with 16,000–18,000 lb on an 8¾-inch bit, the rate of penetration in the anhydrite was about 1·5 ft/hr (half the rate obtained with water-base muds). A 5¼-inch diamond core barrel penetrated the limestone at 2 ft/hr, being slower than with water-base mud, but core recovery was 100 per cent in nearly all cases.

The cores were sealed in tins to avoid evaporation losses. Crushed samples from the cores were extracted with toluene in a modified Dean and Stark apparatus. Consistent results were obtained for the connate water content, even though at times the water content of the oil-base mud was as high as 20 per cent.

EXAMPLE OF INTERPRETATION OF RADIOACTIVITY AND RESISTIVITY LOGS IN A SHALY SAND. P. A. Poupon. (Schlumberger International.)

The earlier empirical formulæ developed for the interpretation of electric logs were applicable only to formations containing little clayey matter. Later methods were proposed which could be used when there was a moderate shale content, provided that mud filtrate penetration was at least 4 inches, that the residual hydrocarbon content of the invaded zone was low, and that the electrofiltration component of the S.P. was low. Some other information was needed in addition.

In the Burgan series, however, the invasion is often very small and/or the residual oil saturation is high, while S.P.

deflections opposite clean formations are only 50–60 per cent of the value predicted by the conventional formula. As a result it does not seem possible to use the Poupon–Loy–Tixier interpretation charts. Gamma ray and neutron logs have therefore been employed.

The gamma ray log is basically a recording of the shale content of the rocks, provided that the shales have a constant amount of radioactive material, and that such material occurs only in the shales. Ideally, if a sufficient number of cores were available, and their shale content could readily be obtained, it would be possible to ascertain whether there was a simple relationship between shale content and gamma ray deflections. Failing this, it seems that in some cases a relationship between shale content and gamma ray deflections can be obtained from the logs.

For the Third Burgan sand, which was water-bearing in a Magwa well, a plot of neutron against gamma ray deflections gave points which were fairly close to the average curve, and it appears that the dominant physical parameter in the relationship is the shale content, because of the parallelism between gamma ray and S.P. curves. When the average

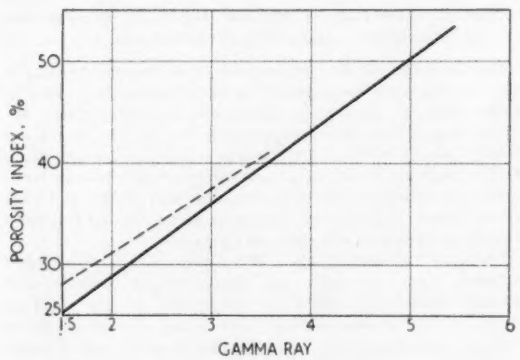

Fig 2

curve was replotted as the neutron-derived porosity index against the gamma ray deflections there was an almost linear relationship. Correction for the high sodium chloride content of the water in the interval studied yielded a relationship shown by the broken curve of Fig 2. This suggests the probability that the gamma ray deflection and the porosity index are approximately linear functions of the percentage of shale.

The porosity index in a shaly formation is the sum of the porosity index corresponding to the effective porosity and that corresponding to the shale content. The latter can be estimated from a knowledge of the shale content (derived from the gamma ray curve) and a factor based on the porosity index for the case where the shale content approaches unity. Fig 2 indicates this factor to be 55–60 per cent.

In order to interpret the true resistivity in terms of fluid saturations, it is necessary to know how the shale is distributed in the sands, because shale laminations will have a different effect from disseminated shale particles.

Accepting the relationship shown by the dashed line of Fig 2, the resistivities of 100 per cent water-bearing shaly sands were calculated as a function of gamma ray deflections for sands with shale laminæ (solid curve, Fig 3) and for sands with disseminated shales (dashed curve, Fig 3). Although the curves do not differ greatly, the dots representing the observations for the Third Burgan sand show better agreement with the solid curve, and hence the sand is inferred probably to have shale laminæ. It is considered that in oil-bearing sands the relationship between gamma ray deflections and resistivity would not reveal so clearly the influence of the shales.

In the absence of an induction log it would be very difficult to reach a conclusion regarding the shale distribution, because the R_t values would not be very accurate.

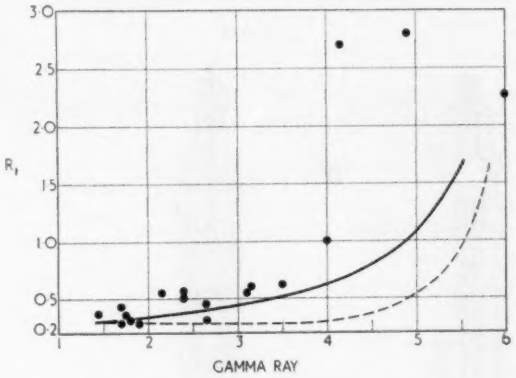

Fig 3

Temperature Notes on the Bahrain Field. W. H. Cotter. (The Bahrain Petroleum Co. Ltd.)

When temperature data for wells completed in the Second Pay limestone of the Bahrain zones were corrected to a datum of 1900 ft subsea and plotted, the contours of equal temperature broadly followed the structure contours, but gave a northerly shift of the temperature "crest" relative to the

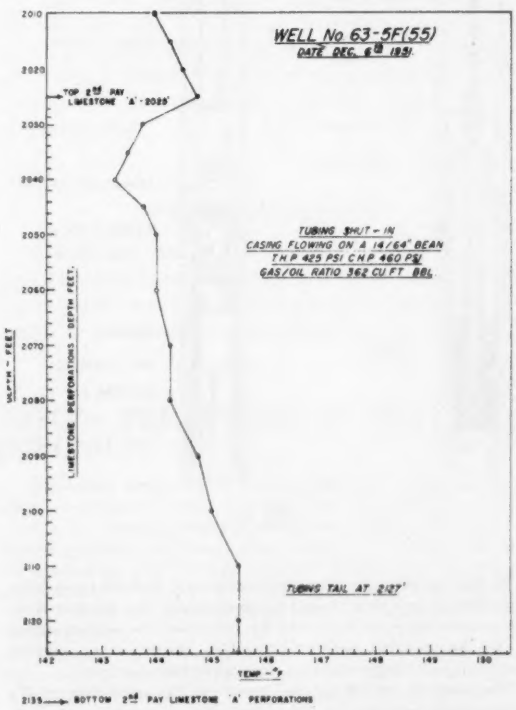
WELL No 63-5F(55)
DATE DEC. 6th 1954
TOP 2nd PAY LIMESTONE 'A' - 2025'
TUBING SHUT-IN
CASING FLOWING ON A 14/64" BEAN
THP 425 PSI CHP 460 PSI
GAS/OIL RATIO 362 CU FT BBL
TUBING TAIL AT 2127'
LIMESTONE PERFORATIONS - DEPTH FEET.
DEPTH - FEET
TEMP - °F
2135 BOTTOM 2nd PAY LIMESTONE 'A' PERFORATIONS

Fig 4

structural crest. From 300 ft subsea, where the temperature is fairly constant at 86° F, to the Bahrain zones, the temperature gradient is about 3·3° F/100 ft.

Wells in the north beyond two faults have water with a much higher sulphate content than those to the south in the same horizon. All waters in the lower pays of the Bahrain zones are rich in sulphate, suggesting that there may be movement of water via the faults to the northern sector. This may be of significance with regard to the temperature "crest" shift.

From the Bahrain zones to the Arab zones the geothermal gradient is about 2° F/100 ft.

Gas injection has been used for pressure maintenance in the Second Pay limestone of the Bahrain zones. A temperature increase of about 2° F has been observed near the topmost perforations (Fig 4) of those wells which have been reached by the gas, the arrival being shown by a sharp rise in gas/oil ratio. This temperature rise occurs after the gas reaches the well, and is attributed to free gas dissolving in the highly undersaturated crude.

A Short and Simple Method of Determining the Salinity of Some Crude Oils. P. Branson. (Qatar Petroleum Co. Ltd.)

Oil with salt content in excess of 10 lb NaCl/100 brl tends to increase corrosion of refinery equipment. A simple test was, therefore, developed for determining the salt content of oil from Qatar. This involves shaking 75 cc of crude with 75 cc of distilled water containing three drops of Lissapol in a separating funnel for 10 minutes. After standing for 10 minutes, 50 cc of water was drawn off and titrated with standard silver nitrate solution.

For salinities below 50 lb NaCl/1000 brl the results were repeatable, and comparable with those obtained by Standard Method IP 77/51, although much lower in absolute accuracy.

The method was not successful with Ain Zalah and Kirkuk crudes, because these gave emulsions from which only a few cc of clear water separated. A de-emulsifier other than Lissapol may be needed for the successful use of this method with these crudes.

GAS–LIQUID CHROMATOGRAPHY: EFFECT OF SUPPORT SIZE AND PROPORTION OF LIQUID PHASE ON COLUMN EFFICIENCY*

By J. D. CHESHIRE and R. P. W. SCOTT†

SUMMARY

This paper describes the effect on efficiency of support size, proportion of liquid phase to support, and length of column. The conditions required for obtaining column efficiencies of 12,000 theoretical plates are given. The effect of size of charge on high-efficiency columns is also discussed.

INTRODUCTION

In their work on column efficiency Keulemans and Kwantes [1] stated that the support size of 25 to 85 B.S. mesh (particle diameter 599 to 178 μ) produced columns of maximum efficiency. They also stated that the particle size affected eddy diffusion occurring in the column and expressed the eddy diffusion effect algebraically by the function, $2\lambda d_p$, originally derived by van Deemter, Zuiderweg, and Klinkenberg,[2] where λ is a characteristic of the packing and d_p is the average particle diameter. This function contributes to the height of the theoretical plate and increases as the overall efficiency of the column decreases. The authors [1] explained that although it appears that a reduction of d_p would increase the overall efficiency of the column, this is not always so. A point would be reached at which further reduction of d_p would produce irregular packing and channelling, resulting in an increase in the factor λ and a corresponding fall in overall efficiency.

The grade of 25 to 85 B.S. mesh, suggested by Keulemans and Kwantes for column supports, has not been used by all workers. In their original work on gas–liquid chromatography, James and Martin [3] used Celite graded to an average particle diameter of 40 μ. Adlard,[4] in his work on the polyethylene glycols, used supports graded to 85 to 120 B.S. mesh (178 to 124 μ), and Mellor [5] stated broadly that the efficiency of a column increased as the particle diameter of the support was reduced.

Widely differing quantities of liquid phase on the support have also been reported. Keulemans and Kwantes [1] state that too high a proportion of liquid phase to support affects the mass transfer taking place in the column and reduces the overall efficiency. Too small a ratio of liquid phase, however, tends to introduce adsorption problems in connexion with the support itself. They have effected a compromise and state that the percentage of liquid phase on the support should be between 13 and 17 per cent w/w. Grant and Vaughan [6] and Brooks, Murray, and Williams [7] used 23·1 per cent w/w liquid phase on support. James and Martin,[3] Harrison,[8] and Adlard [4] all used 28·6 per cent w/w liquid phase on support, while Keppler, Dijkstra, and Schols [9] in their work at high temperatures used between 9 and 33 per cent w/w liquid phase on the support.

Thus an examination of the literature leads to the conclusions that there is still some uncertainty as to the best particle size to employ for the support and that there is little agreement on the quantity of liquid phase to be used on the support, values from 9 to 33 per cent w/w having been reported. The work described in this paper was carried out to improve the separation of hydrocarbons. The correct particle size to be used for the support and the optimum proportion of liquid phase to support that is required to produce the maximum column efficiency were determined.

PARTICLE SIZE OF SUPPORT

Experimental

Previous work had shown that with the particular method of packing used, Johns–Manville C22 insulating brick was the most suitable material for the support in gas–liquid chromatography. These bricks were ground, sieved to the following mesh sizes, and treated with 20 per cent Apiezon oil A as a liquid phase.

Support grades	Particle diameters
30 to 60 B.S. mesh	500 to 251 μ
60 to 100 ,,	251 to 152 μ
100 to 120 ,,	152 to 124 μ
120 to 160 ,,	124 to 95 μ
100 to 200 ,,	152 to 76 μ

The apparatus used was the routine equipment previously described by Scott,[10] and the column was packed by vibration obtained from a low-speed elec-

* MS received 15 November 1957.

† Benzole Producers Ltd.

tric motor. The column conditions used were as follows:

Column length	5 ft
Column diameter	3·6 mm
Operating temperature	78° C
Carrier gas	25% nitrogen, 75% hydrogen
Carrier-gas flow	10 to 65 ml/min
Column charge	3 to 3·5 mg

Chromatographs were obtained for *n*-heptane using a series of gas flows for each grade of support and the column efficiency [11] calculated. The results are shown in Fig 1.

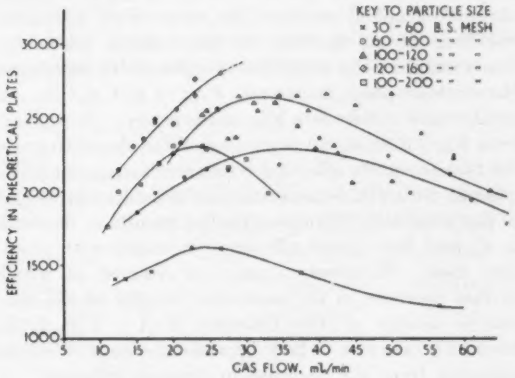

FIG 1

EFFICIENCIES FOR *n*-HEPTANE ON SUPPORTS OF VARIOUS PARTICLE SIZE

Discussion

Examination of Fig 1 shows that the smaller the particle size of the support, the higher the resulting column efficiency at the optimum gas flow, which agrees with the results obtained by Mellor.[5] The optimum flow could not be reached for support graded 120 to 160 B.S. mesh, as the pressure required to ensure the necessary flow of gas through the column was too high for the safe use of the glass injection system. It should be noted that the wide-range support grade, 100 to 200 B.S. mesh, gives lower efficiencies than the narrow-range support grades, 100 to 120 and 120 to 160 B.S. mesh. The results show that if the pressure at the inlet of the column is limited to a value that is safe for glass apparatus, the optimum size of support is the narrow grade of 100 to 120 B.S. mesh.

QUANTITY OF LIQUID PHASE ON THE SUPPORT

Experimental

The apparatus was the same as that used in the investigation of optimum particle size. The column conditions were as follows:

Column length	5 ft
Column diameter	3·6 mm
Operating temperature	78° C
Carrier gas	25% nitrogen, 75% hydrogen
Carrier-gas flow	6 to 65 ml/min
Support	Johns–Manville C22 insulating brick
Grade of support	100 to 120 B.S. mesh
Liquid phase	Apiezon oil A
Column charge	0·2 to 3·5 mg

A mixture of *n*-pentane, *n*-hexane, and *n*-heptane was chromatographed at various gas flows and the optimum flow, which gave the maximum column efficiency, was determined for columns carrying 2·5, 5, 10, 15, 20, 25, and 30 per cent w/w of liquid phase on the support. A chromatogram of the following hydrocarbon mixture was then obtained for each proportion of liquid phase to support using the optimum flow previously determined:

n-pentane
n-hexane
n-heptane
n-octane
methyl*cyclo*hexane
benzene
toluene
ethylbenzene
o-xylene
p-xylene
n-propylbenzene
*iso*propylbenzene

A graph of the efficiencies obtained plotted against retention time for each proportion of liquid phase to support is shown in Fig 2.

Discussion

The results shown in Fig 2 indicate that, when employing a 5-ft column, 3·6 mm in diameter, to chromatograph substances that boil in the neighbourhood of the column temperature, the optimum quantity of liquid phase to support that gives the maximum column efficiency is 20 per cent w/w. If the column is used at the optimum gas flow, this quantity of liquid phase will produce efficiencies of the order of 2500 to 3500 theoretical plates. Such efficiencies will be obtained for substances with retention times lying between 5 and 60 minutes.

The shapes of the curves in Fig 2 are also of considerable interest. Confining attention to curves for 10 per cent or more of liquid phase to support, the efficiency increases rapidly with retention time during the first 5 minutes of the chromatogram. After this point the slope of the efficiency/retention time curve decreases suddenly, and, for a support carrying 30 per cent w/w of liquid phase, the efficiency after 25 minutes shows very small increase as the retention

time increases. The following equation, derived by van Deemter, Zuiderweg, and Klinkenberg,[2] and used by Keulemans and Kwantes,[1] gives algebraic representation of all of the factors affecting column efficiency:

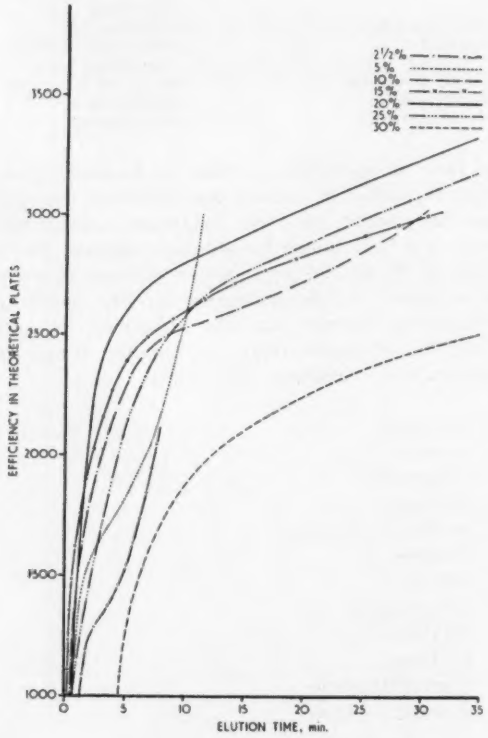

FIG 2

EFFECT OF PROPORTION OF LIQUID PHASE ON EFFICIENCY

$$H = 2\lambda d_p + \gamma D_g/u + 8/\pi^2 \cdot k'/(1+k')^2 \cdot d_f^2/D_l \cdot u$$

where H = height equivalent to a theoretical plate and, for a column of a given length, is inversely proportional to efficiency;

λ = a characteristic of the packing;

d_p = average particle diameter;

γ = a correction factor for the tortuosity of gas path;

D_g = diffusivity in gas phase;

D_l = diffusivity in liquid phase;

u = linear velocity of gas;

$k' = K \cdot F_l/F_g$, where K = partition coefficient of solute and F_l and F_g = the fractions of cross section occupied by the liquid and gas phases respectively;

d_f = effective thickness of liquid film on the support.

As mentioned before, the function $2\lambda d_p$ accounts for the eddy diffusion occurring in the column. The function $\gamma D_g/u$ accounts for the gaseous or molecular diffusion occurring in the column and

$$8/\pi^2 \cdot k/(1+k')^2 \cdot d_f^2/D_l$$

accounts for the resistance to mass transfer.

Under the conditions in which the results in Fig 2 were obtained, λd_p and u were constant and, for a given curve, D_g, D_l, and $k'/(1+k')^2$ were the only variables. The factor D_g is associated with the gaseous diffusion effect only, and D_l and $k'/(1+k')^2$ are associated with the resistance to mass transfer only. As the molecular weight, and thus the partition coefficient and retention time, of the substances chromatographed increase, the value of D_g decreases, resulting in an increase in the column efficiency. Conversely, as the molecular weight of the substance chromatographed increases, $k'/(1+k')^2 \cdot d_f^2/D_l$ increases and results in a loss of efficiency. It appears from Fig 2 that at the beginning of the chromatogram the fact adversely affecting column efficiency is mainly gaseous diffusion, because increase in molecular weight of the substance chromatographed results in decrease in D_g and the column efficiency increases with retention time. However, a point is reached at which further increase in the molecular weight of the substance results in the function $k'/(1+k')^2 \cdot d_f^2/D_l$ becoming effective, which opposes the improvements resulting from the decrease in gaseous diffusion. It is concluded therefore that the change of slope in the efficiency/retention time curve occurs where the rate of change of resistance to mass transfer becomes of the same order as the rate of change of gaseous diffusion, and the rate of increase in column efficiency is thus reduced. The almost horizontal part of the curve for 30 per cent w/w liquid phase on support indicates that the increase in efficiency due to the reduction of gaseous diffusion is approximately equal to the increase in resistance due to mass transfer. However, this sudden change of slope does not occur so early in the elution time curve, when 5 per cent w/w or less of liquid phase is present on the support. There is little or no change in the slope of the efficiency/retention time curve for elution times up to 12 minutes, and the efficiency continues to increase rapidly. This would be expected, as under these conditions d_f^2 becomes so small that the resistance to mass transfer factor no longer becomes effective. Although substances with elution times greater than 12 minutes could not be placed on the column due to low volatility at the operating temperature, it is obvious that this sudden change of slope would eventually occur if longer retention times could be realized. The effect of mass transfer has been considerably reduced, but, of course, cannot be completely eliminated. These results lead to two conclusions:

(1) High-efficiency columns for chromatographing high-boiling liquids can best be pre-

pared by using short columns run at a low temperature with a low proportion of liquid phase to support.

(2) High-efficiency columns for chromatographing low-boiling substances may be obtained by increasing the length of the column to give a longer chromatogram, but, at the same time, maintaining the same proportion of liquid phase to support, namely, 5 per cent w/w.

CONDITIONS FOR HIGH EFFICIENCY

Experimental

To verify these conclusions, the following columns were constructed and their efficiencies determined:

	Column 1	Column 2	Column 3
Length	5 ft	10 ft	25 ft
Diameter	3·6 mm	3·6 mm	3·6 mm
Operating temperature	101° C	78° C	78° C
Carrier gas	25% N_2/75% H_2	25% N_2/75% H_2	25% N_2/75% H_2
Carrier-gas flow	37 ml/min	37 ml/min	37 ml/min
Support	Johns–Manville C22 insulating brick	Johns–Manville C22 insulating brick	Johns–Manville C22 insulating brick
Grade of support	100–120 B.S. mesh	100–120 B.S. mesh	60–100 B.S. mesh
Liquid phase	Apiezon oil A	Apiezon oil A	Apiezon oil A
% Liquid phase to support	5% w/w	5% w/w	5% w/w
Column charge	0·2–0·7 mg	0·2–0·8 mg	0·4 mg
Inlet pressure	20 lb/sq. in.	22 lb/sq. in.	35 lb/sq. in.

The support grade of 60 to 100 B.S. mesh was used for column 3 to reduce the back pressure at the column inlet. The injection system on column 3 was also modified to ensure safe use at the high inlet-pressure.

These columns were used to chromatograph the following mixtures:

Mixture 1		Mixture 2		Mixture 3	
Substance	Boiling point °C	Substance	Boiling point °C	Substance	Boiling point °C
n-Pentane	36·0	*n*-Hexane	68·8	*n*-Pentane	36·0
n-Hexane	68·8	Benzene	80·1	*n*-Hexane	68·8
Benzene	80·1	*n*-Heptane	98·4	Methylcyclopentane	71·8
n-Heptane	98·4	Methylcyclohexane	101·0	Benzene	80·1
Methylcyclohexane	101·0	Toluene	110·8	*n*-Heptane	98·4
Toluene	110·8	Ethylbenzene	136·2	Methylcyclohexane	101·0
n-Octane	125·6	*p*-Xylene	138·4	Toluene	110·8
Ethylbenzene	136·2	*o*-Xylene	144·0	*n*-Octane	125·6
p-Xylene	138·4	isoPropylbenzene	152·4	Ethylbenzene	136·2
o-Xylene	144·0	*n*-Propylbenzene	158·6	*m*-Xylene	137·0
isoPropylbenzene	152·4	Mesitylene	164·6	*p*-Xylene	138·4
n-Propylbenzene	158·6	*p*-Cymene	177·3	*o*-Xylene	144·0
Mesitylene	164·6				
n-Decane	174·0				
Hydrindene	178·0				
Indene	182·0				
n-Undecane	195·8				

The optimum flow that gave the maximum efficiency was found to be approximately 37 ml/min for all three columns. Mixtures 1, 2, and 3 were chromatographed on columns 1, 2, and 3 respectively. The chromatogram obtained from column 3 is shown in Fig 3. The efficiency for each substance in all three chromatograms was calculated and the efficiencies, plotted against retention time, are shown in Fig 4. It should be stressed that the size of the column charge became very critical when using column 3. Previous work had shown that when using 20 per cent w/w liquid phase on the support, the column efficiency was not affected by the quantity of the charge, providing it was kept below 4 mg. However, when using columns 1 and 2 it was found that if the total charge exceeded 1 mg asymmetric peaks were formed and poor efficiency resulted. This effect was even more pronounced with column 3, for which a total charge in excess of 0·4 mg resulted in asymmetric peaks. This corresponded to a maximum of about 0·08 mg for any individual component present in the mixture. Due to the necessity for maintaining low column-charges, the sensitivity of the detector used in this work will be of interest and is stated as follows:

A hydrocarbon produced 2 per cent of full-scale deflection of the recorder at a concentration of 0·05 μg per ml of carrier gas. This sensitivity was obtained with a long-term instability or drift of <1 per cent full-scale deflection and a short-term instability or noise of $<0·3$ per cent full-scale deflection.

Discussion

It may be seen from Fig 4 that a change of slope occurs in the efficiency/retention time curve obtained for all three columns, but that this inflection occurs at progressively higher efficiency values as the length of the column is increased. A 5-ft column run at 101° C may be used for chromatographing substances boiling up to 200° C with a column efficiency of up to 4000 theoretical plates and a total elution time of 40 minutes. A 25-ft column run at 78° C will produce chromatograms for substances boiling up to 144° C with an efficiency of about 12,000 theoretical plates and a total elution time of 120 minutes. Efficiencies of 8000 to 9000 plates were obtained from this column

TABLE 1

Substance	Efficiency in theoretical plates
n-Hexane	3620
Benzene	3090
n-Heptane	4210
Methylcyclohexane	4560
Toluene	4340
n-Octane	5440
Ethylbenzene	4490
p-Xylene	4370
m- and *p*-Xylene	3160
o-Xylene	4980
Cumene	5250
n-Propylbenzene	5360
Mesitylene	5060
p-Cymene	6520

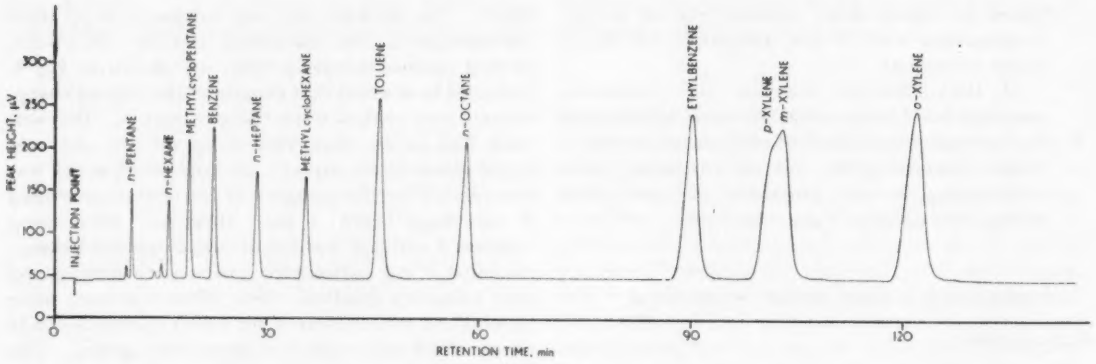

FIG 3

CHROMATOGRAM OF A HYDROCARBON MIXTURE

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CONCLUSIONS

The particle size of the support that gives the highest column-efficiency commensurate with reasonably low column inlet-pressures is the close-cut grade of 100 to 120 B.S. mesh. The adverse effect of liquid diffusion on column efficiency may be reduced by using 5 per cent w/w or less of liquid phase to support and, at the same time, increasing the column length to obtain practical retention values. A column of 10,000 to 12,000 theoretical plates, suitable for the separation of hydrocarbons, may be obtained from a column 25 ft long, 3·6 mm in diameter, and packed with 5 per cent w/w of Apiezon oil on Johns–Manville C22 insulating brick graded to 60 to 100 B.S. mesh.

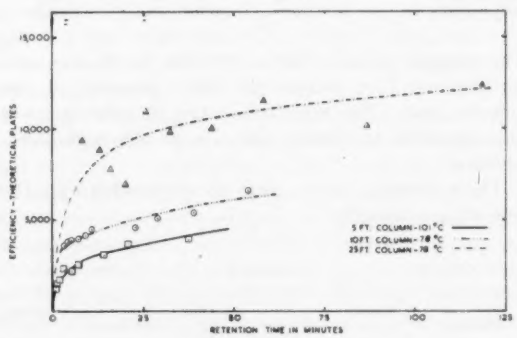

FIG 4

EFFECT OF RETENTION TIME AND COLUMN LENGTH ON EFFICIENCY

To obtain these high efficiencies, the total charge on the column must be less than 0·4 mg and a suitable detector must be employed with sufficiently high sensitivity and stability to produce the required chromatograms.

ACKNOWLEDGMENTS

The authors wish to thank K. A. Knight for his valuable assistance and the directors of Benzole Producers Ltd for permission to publish this work.

References

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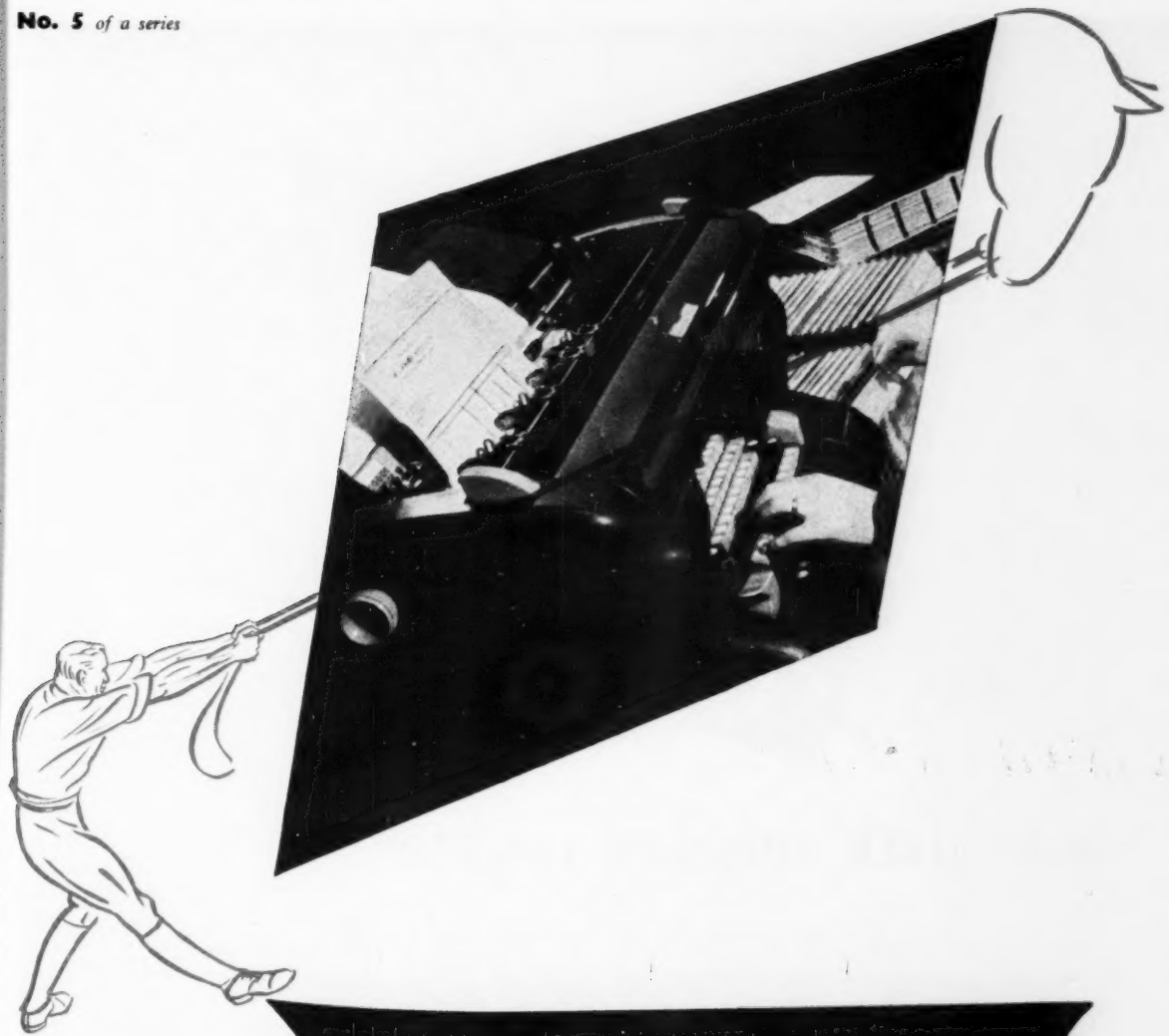

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time increases. The following equation, derived by van Deemter, Zuiderweg, and Klinkenberg,[2] and used by Keulemans and Kwantes,[1] gives algebraic representation of all of the factors affecting column efficiency:

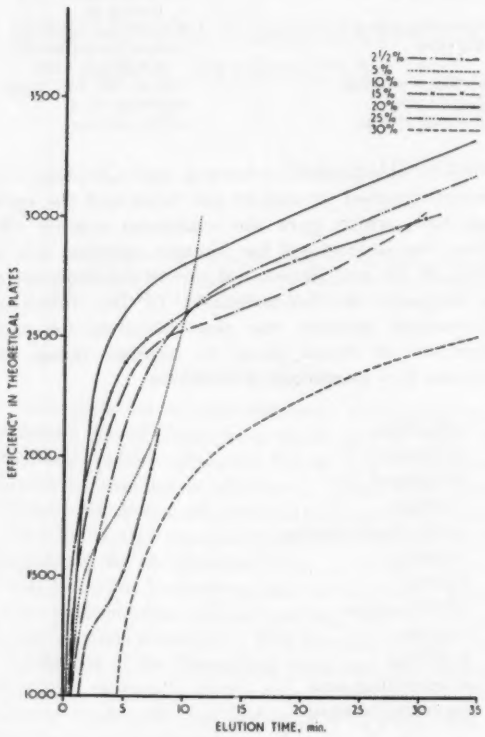

FIG 2

EFFECT OF PROPORTION OF LIQUID PHASE ON EFFICIENCY

$$H = 2\lambda d_p + \gamma D_g/u + 8/\pi^2 \cdot k'/(1+k')^2 \cdot d_f^2/D_l \cdot u$$

where H = height equivalent to a theoretical plate and, for a column of a given length, is inversely proportional to efficiency;

λ = a characteristic of the packing;

d_p = average particle diameter;

γ = a correction factor for the tortuosity of gas path;

D_g = diffusivity in gas phase;

D_l = diffusivity in liquid phase;

u = linear velocity of gas;

$k' = K \cdot F_l/F_g$, where K = partition coefficient of solute and F_l and F_g = the fractions of cross section occupied by the liquid and gas phases respectively;

d_f = effective thickness of liquid film on the support.

As mentioned before, the function $2\lambda d_p$ accounts for the eddy diffusion occurring in the column. The function $\gamma D_g/u$ accounts for the gaseous or molecular diffusion occurring in the column and

$$8/\pi^2 \cdot k/(1+k')^2 \cdot d_f^2/D_l$$

accounts for the resistance to mass transfer.

Under the conditions in which the results in Fig 2 were obtained, λd_p and u were constant and, for a given curve, D_g, D_l, and $k'/(1+k')^2$ were the only variables. The factor D_g is associated with the gaseous diffusion effect only, and D_l and $k'/(1+k')^2$ are associated with the resistance to mass transfer only. As the molecular weight, and thus the partition coefficient and retention time, of the substances chromatographed increase, the value of D_g decreases, resulting in an increase in the column efficiency. Conversely, as the molecular weight of the substance chromatographed increases, $k'/(1+k')^2 \cdot d_f^2/D_l$ increases and results in a loss of efficiency. It appears from Fig 2 that at the beginning of the chromatogram the fact adversely affecting column efficiency is mainly gaseous diffusion, because increase in molecular weight of the substance chromatographed results in decrease in D_g and the column efficiency increases with retention time. However, a point is reached at which further increase in the molecular weight of the substance results in the function $k'/(1+k')^2 \cdot d_f^2/D_l$ becoming effective, which opposes the improvements resulting from the decrease in gaseous diffusion. It is concluded therefore that the change of slope in the efficiency/retention time curve occurs where the rate of change of resistance to mass transfer becomes of the same order as the rate of change of gaseous diffusion, and the rate of increase in column efficiency is thus reduced. The almost horizontal part of the curve for 30 per cent w/w liquid phase on support indicates that the increase in efficiency due to the reduction of gaseous diffusion is approximately equal to the increase in resistance due to mass transfer. However, this sudden change of slope does not occur so early in the elution time curve, when 5 per cent w/w or less of liquid phase is present on the support. There is little or no change in the slope of the efficiency/retention time curve for elution times up to 12 minutes, and the efficiency continues to increase rapidly. This would be expected, as under these conditions d_f^2 becomes so small that the resistance to mass transfer factor no longer becomes effective. Although substances with elution times greater than 12 minutes could not be placed on the column due to low volatility at the operating temperature, it is obvious that this sudden change of slope would eventually occur if longer retention times could be realized. The effect of mass transfer has been considerably reduced, but, of course, cannot be completely eliminated. These results lead to two conclusions:

(1) High-efficiency columns for chromatographing high-boiling liquids can best be pre-

pared by using short columns run at a low temperature with a low proportion of liquid phase to support.

(2) High-efficiency columns for chromatographing low-boiling substances may be obtained by increasing the length of the column to give a longer chromatogram, but, at the same time, maintaining the same proportion of liquid phase to support, namely, 5 per cent w/w.

CONDITIONS FOR HIGH EFFICIENCY

Experimental

To verify these conclusions, the following columns were constructed and their efficiencies determined:

	Column 1	Column 2	Column 3
Length	5 ft	10 ft	25 ft
Diameter	3·6 mm	3·6 mm	3·6 mm
Operating temperature	101° C	78° C	78° C
Carrier gas	25% N_2/75% H_2	25% N_2/75% H_2	25% N_2/75% H_2
Carrier-gas flow	37 ml/min	37 ml/min	37 ml/min
Support	Johns–Manville C22 insulating brick	Johns–Manville C22 insulating brick	Johns–Manville C22 insulating brick
Grade of support	100–120 B.S. mesh	100–120 B.S. mesh	60–100 B.S. mesh
Liquid phase	Apiezon oil A	Apiezon oil A	Apiezon oil A
% Liquid phase to support	5% w/w	5% w/w	5% w/w
Column charge	0·2–0·7 mg	0·2–0·8 mg	0·4 mg
Inlet pressure	20 lb/sq. in.	22 lb/sq. in.	35 lb/sq. in.

The support grade of 60 to 100 B.S. mesh was used for column 3 to reduce the back pressure at the column inlet. The injection system on column 3 was also modified to ensure safe use at the high inlet-pressure.

These columns were used to chromatograph the following mixtures:

Mixture 1		Mixture 2		Mixture 3	
Substance	Boiling point ° C	Substance	Boiling point ° C	Substance	Boiling point ° C
n-Pentane	36·0	*n*-Hexane	68·8	*n*-Pentane	36·0
n-Hexane	68·8	Benzene	80·1	*n*-Hexane	68·8
Benzene	80·1	*n*-Heptane	98·4	Methyl*cyclo*pentane	71·8
n-Heptane	98·4	Methyl*cyclo*hexane	101·0	Benzene	80·1
Methyl*cyclo*hexane	101·0	Toluene	110·8	*n*-Heptane	98·4
Toluene	110·8	Ethylbenzene	136·2	Methyl*cyclo*hexane	101·0
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A hydrocarbon produced 2 per cent of full-scale deflection of the recorder at a concentration of 0·05 μg per ml of carrier gas. This sensitivity was obtained with a long-term instability or drift of <1 per cent full-scale deflection and a short-term instability or noise of $<0·3$ per cent full-scale deflection.

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It may be seen from Fig 4 that a change of slope occurs in the efficiency/retention time curve obtained for all three columns, but that this inflection occurs at progressively higher efficiency values as the length of the column is increased. A 5-ft column run at 101° C may be used for chromatographing substances boiling up to 200° C with a column efficiency of up to 4000 theoretical plates and a total elution time of 40 minutes. A 25-ft column run at 78° C will produce chromatograms for substances boiling up to 144° C with an efficiency of about 12,000 theoretical plates and a total elution time of 120 minutes. Efficiencies of 8000 to 9000 plates were obtained from this column

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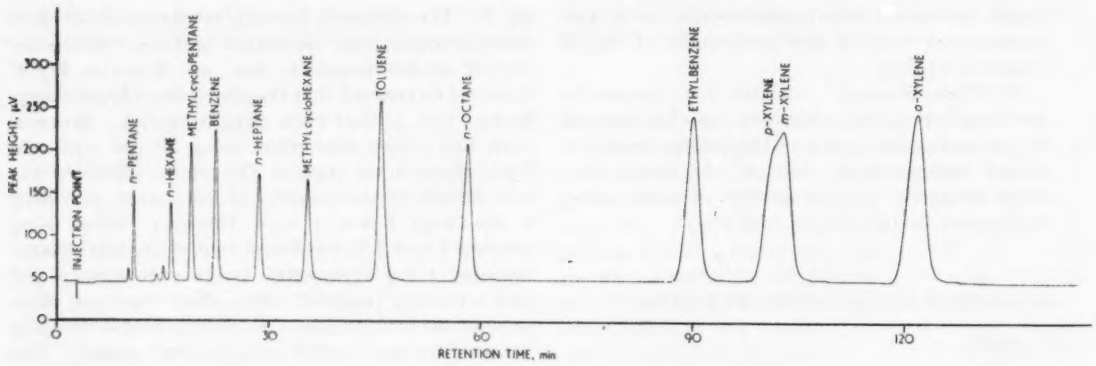

FIG 3

CHROMATOGRAM OF A HYDROCARBON MIXTURE

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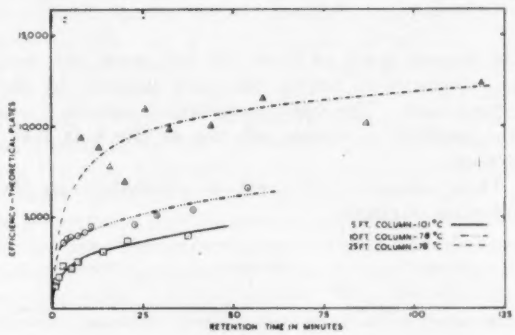

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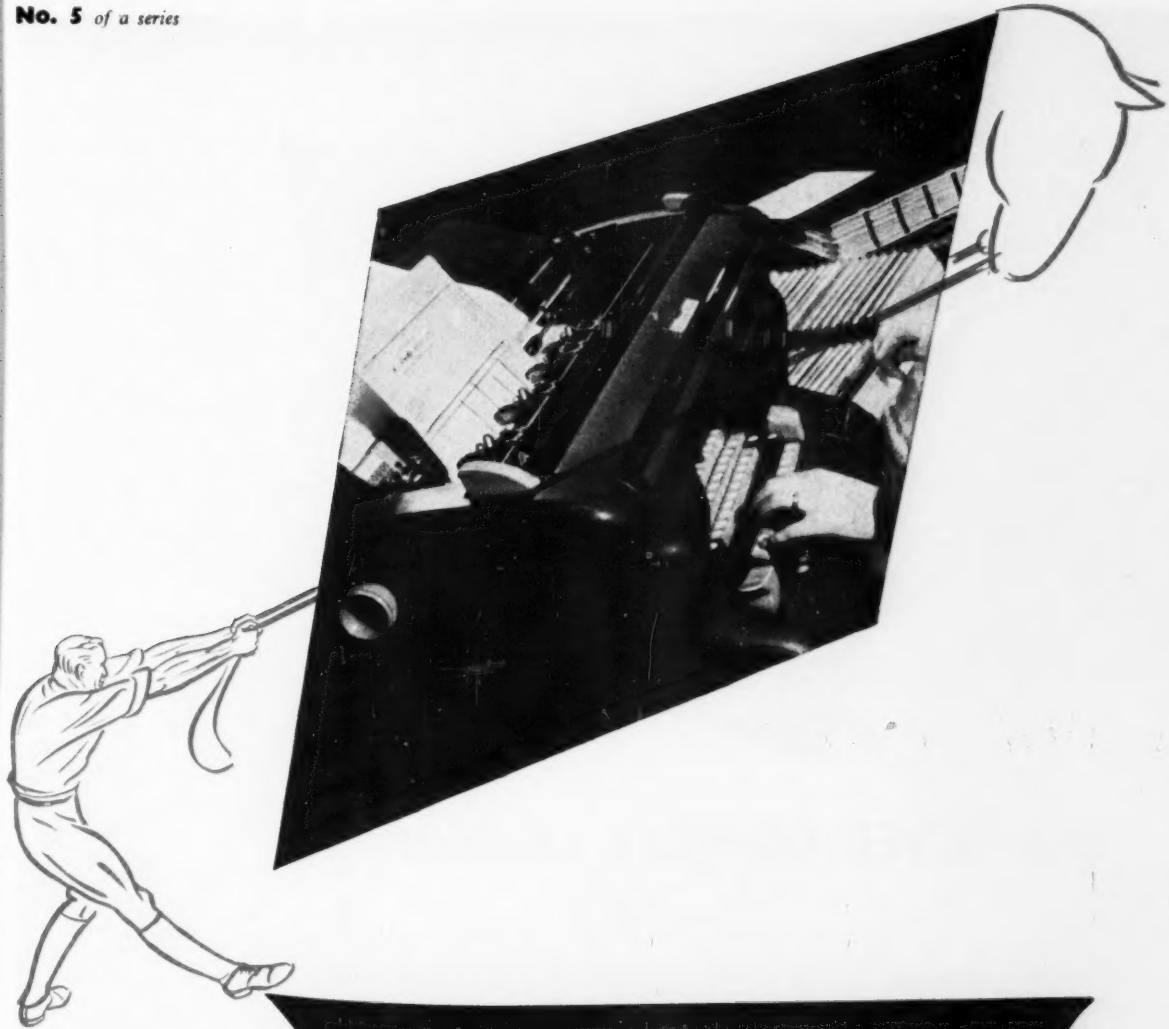

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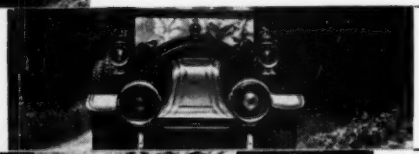
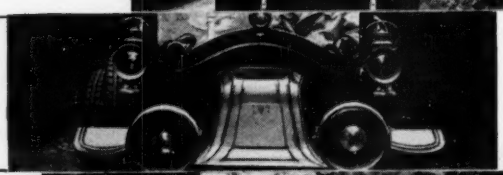
this Filter talk will drive me round the bend!

Sorry, old fruit. I was only trying to be helpful.

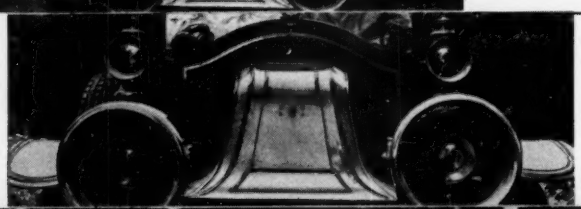
Helpful! Here am I practising for the only real sporting event of the year and all you can talk about is some wretched little gadget on one of your blasted hairyplanes. Can't you see I need to concentrate?



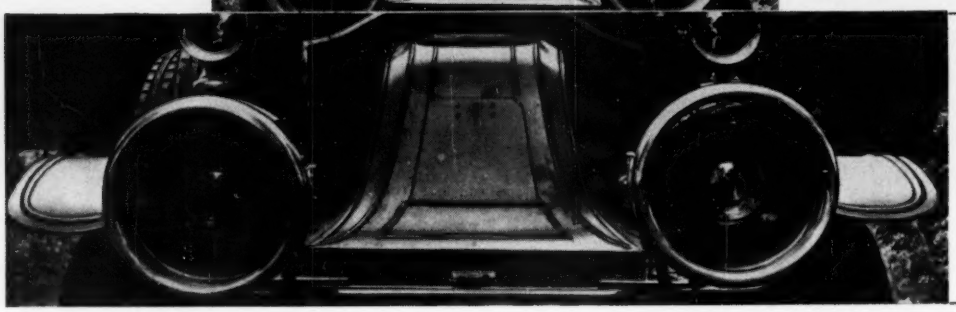
It's not a little gadget and they'd never put it on a plane—it's bigger than this Renault. They put it in the line between the storage and aircraft tanks to make sure the plane gets clean, water-free fuel.



That's the trouble with your infantile modern engines—they have to be fed on strained foods or they get indigestion. Now look at Dotty . . .



Exactly. Do you really think she enjoys having that steam pouring through her exhaust? Now if you took a Simmonds Fram Separator Filter in tow you could have 100% water separation and complete filtration of solids down to 5 microns.



It's tempting, very tempting . . . but it wouldn't be fair on the other competitors. Now d'you mind if we stop a minute . . . I think I really will have to get the rest of that bird's nest out of the tank.

the point of the argument... **FRAM SEPARATOR FILTERS** for clean water-free fuel

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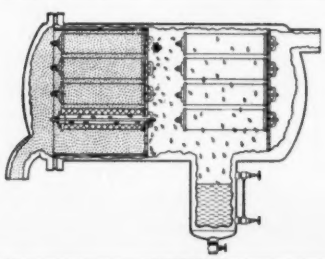

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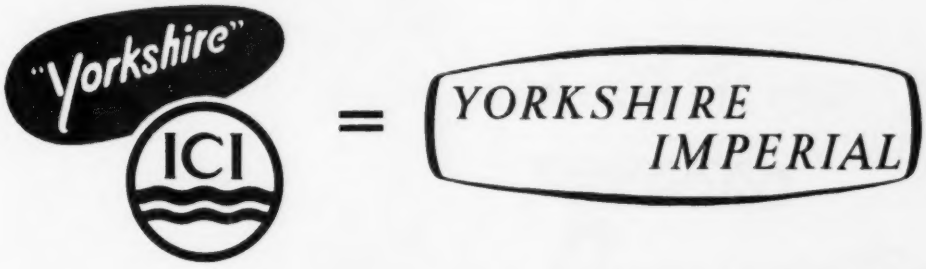

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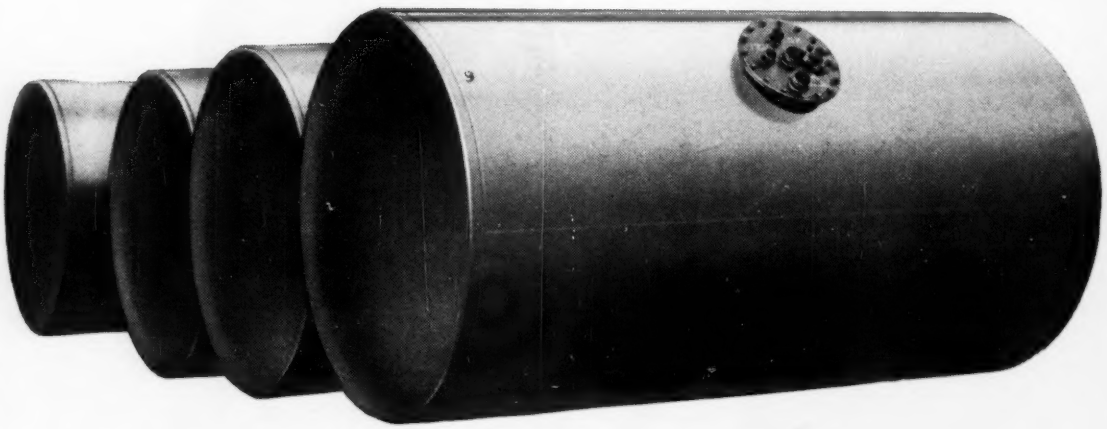

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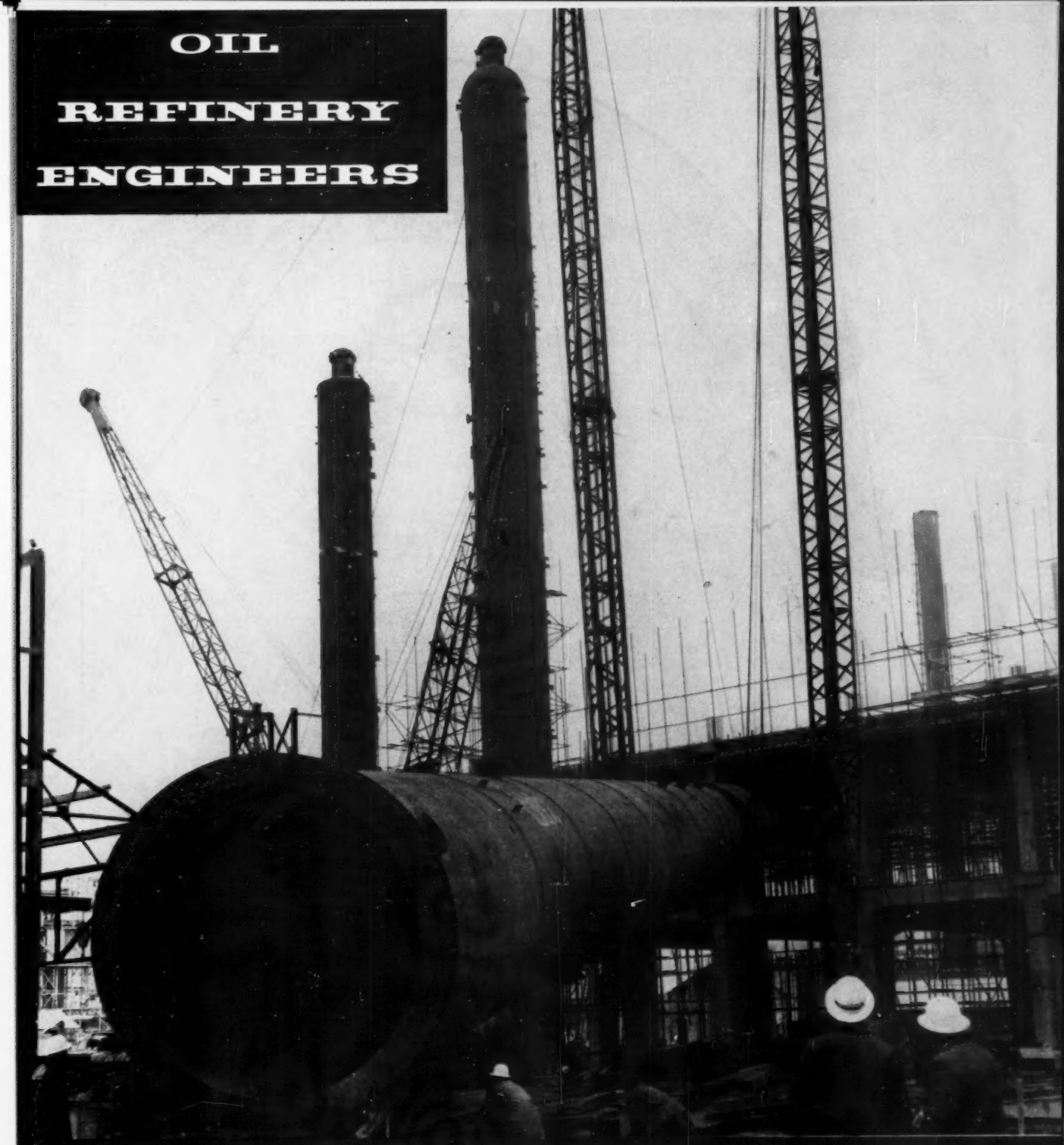

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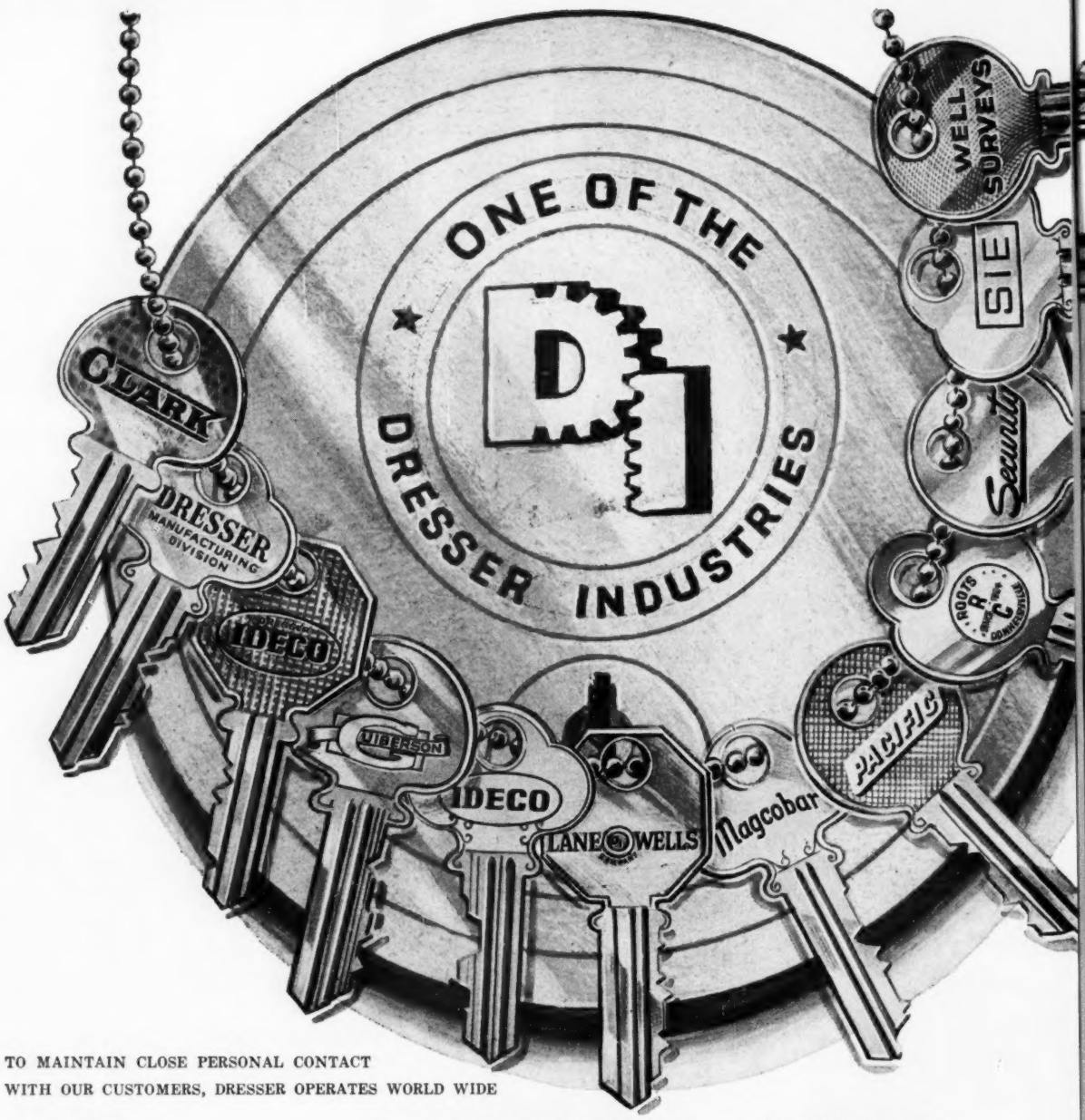

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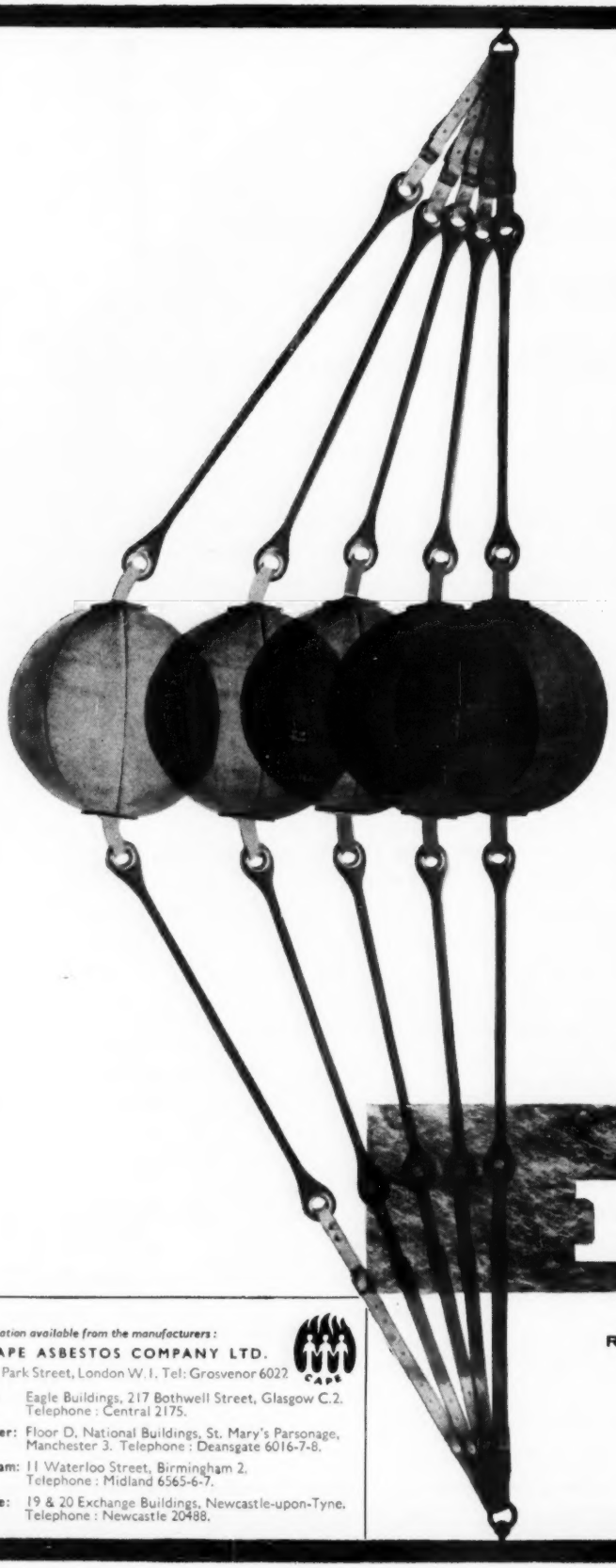

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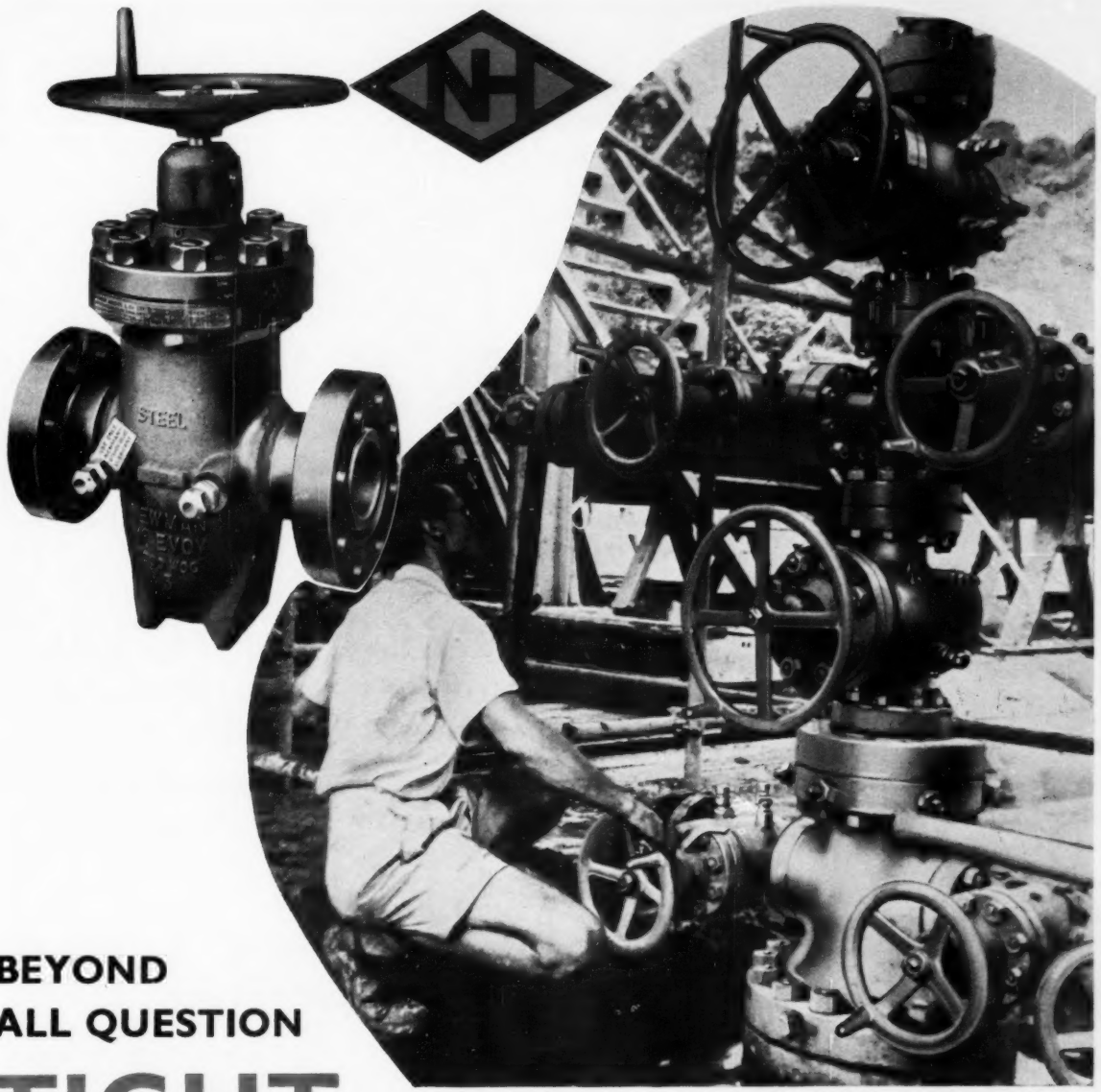

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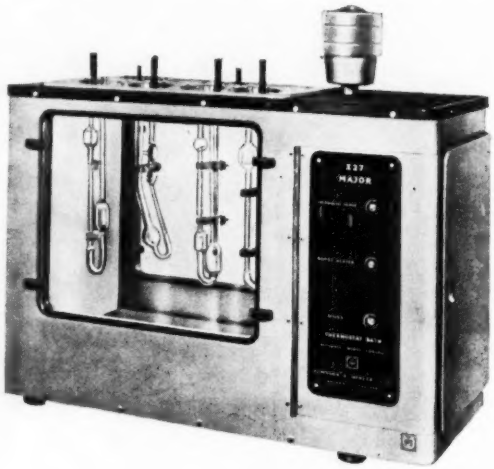

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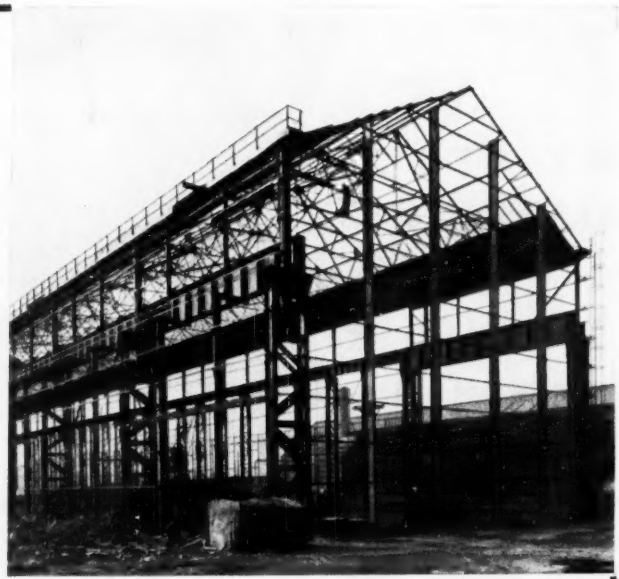

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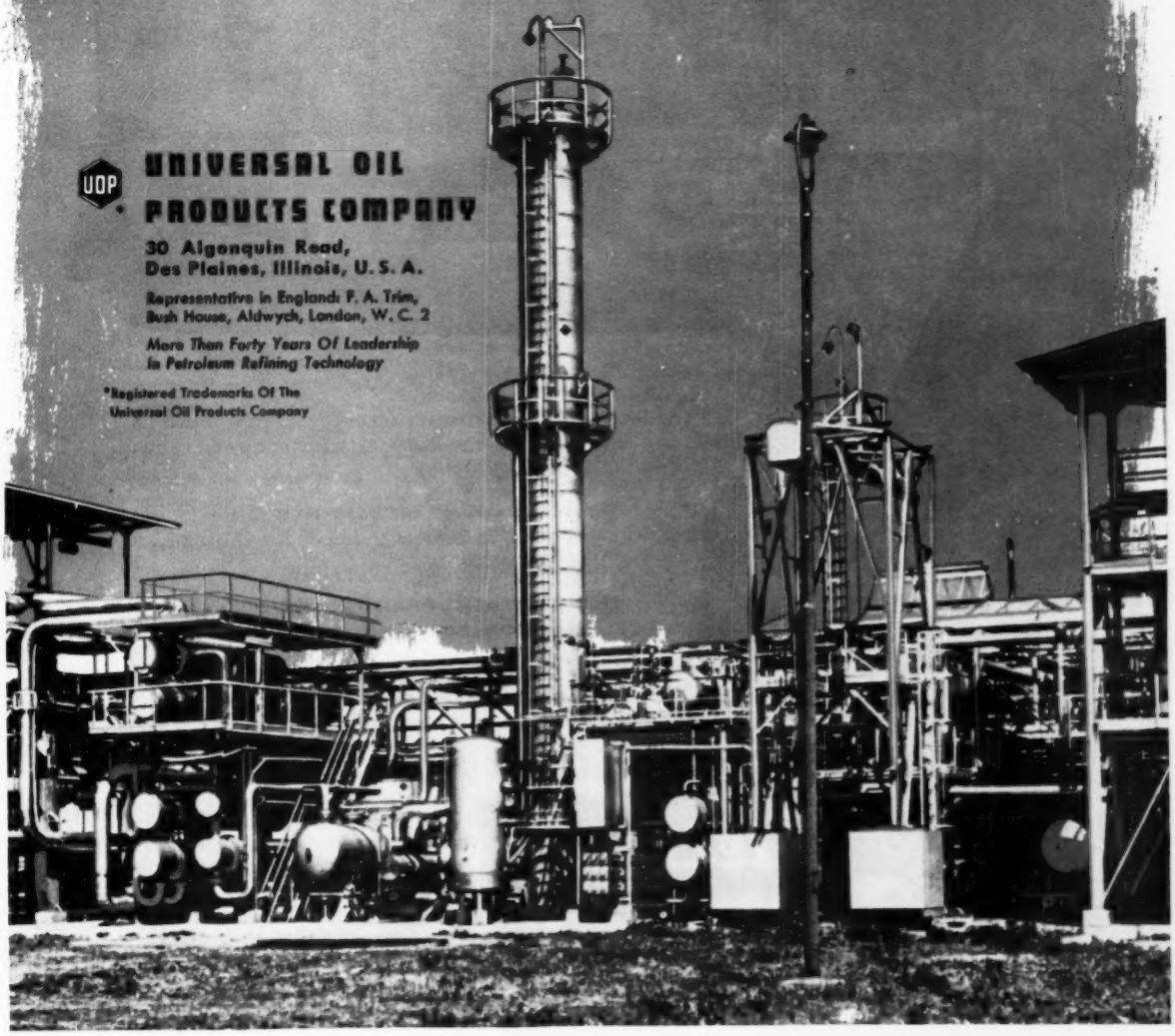

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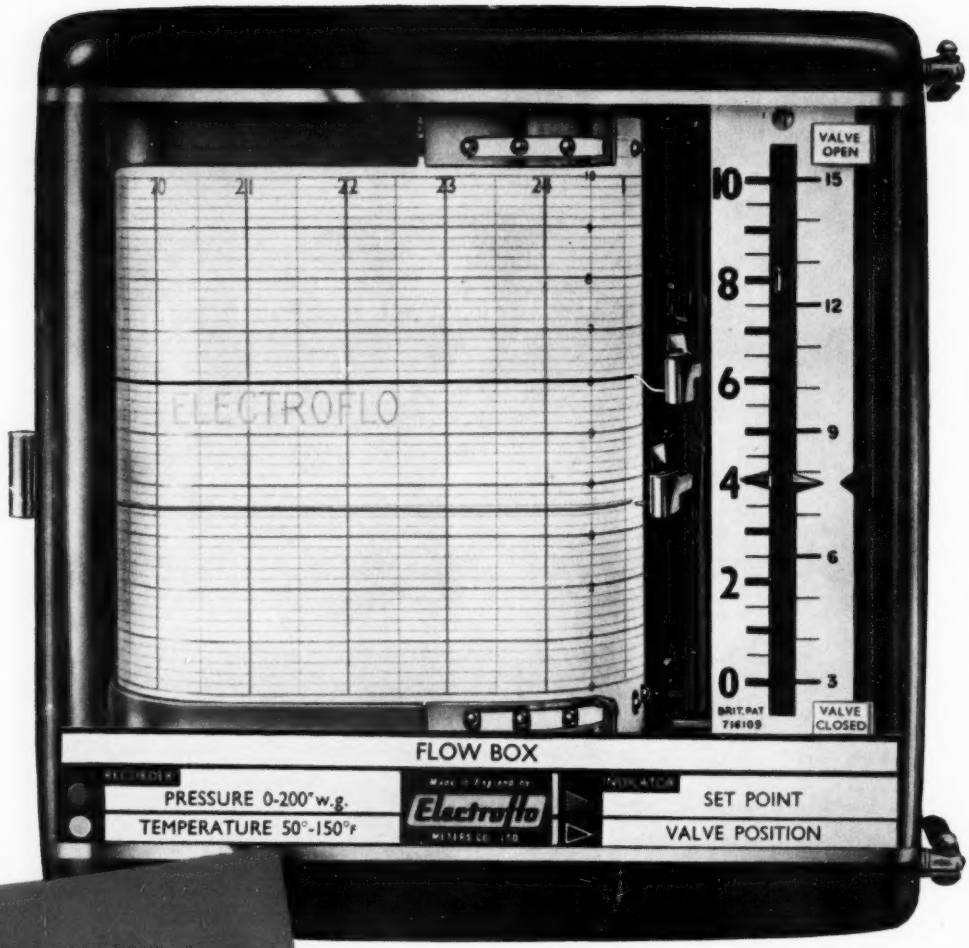

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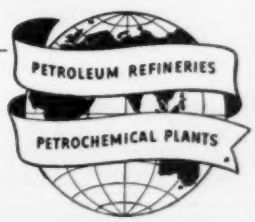

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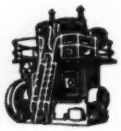

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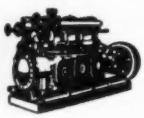

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made for I.C.I. Billingham Division. Length 30 feet. Diameter 10 feet. Fabricated from Kynal M.35/1. Aluminium Alloy $\frac{3}{8}$" thick. One end dished and a rolled body cone and skirt. Argon Arc welded throughout.

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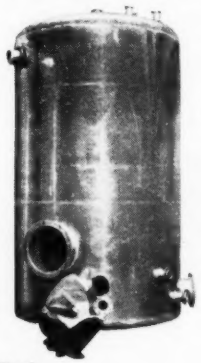
Marston's have unrivalled experience in the fabrication of light alloys—of all shapes and sizes. Their products have earned a reputation for efficiency and reliability that is world-wide.

- ★ *Light Alloy Fabrication*
- ★ *Specialised Engineering Assemblies*
- ★ *Laminated Plastic Components*
- ★ *Flexible Tanks*
- ★ *Radiators and Heat Exchangers*

Tubular Heat Exchangers

5 pass cross contra type made for I.C.I. Billingham Division. Length 18½ feet. Diam. 23½" inside. Header tanks are Aluminium Castings of BS.1490 LM.20 alloy $\frac{3}{8}$" thick. Body rolled and welded from PA.19 Aluminium Alloy Plate $\frac{1}{4}$" thick.
Tested to 75 p.s.i.



Oil Tank

of 2500 gallon capacity, made for I.C.I. Paints Division. Length 14 feet. Diameter 7 feet. Fabricated from Kynal P3. Aluminium alloy $\frac{3}{8}$" thick with dished ends and a rolled plate body. Argon Arc welded throughout. Tested to withstand an 18 foot head of water.

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45 cubic metre Pressure Vessel

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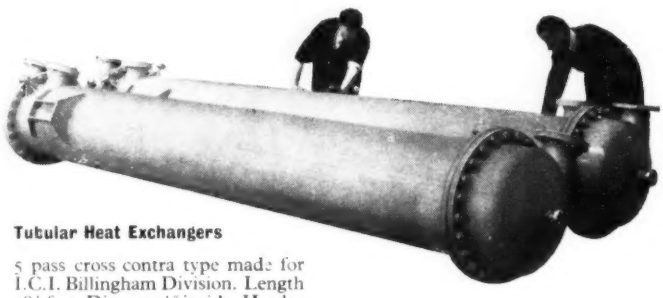

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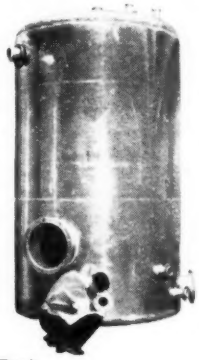

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